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GUIDELINES FOR DEVELOPMENT OF A QUALITY ASSURANCE PROGRAM: VOLUME VIII - DETERMINATION OF CO EMISSIONS FROM STATIONARY SOURCES BY NDIR SPECTROMETRY

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1. Good operating practices
2. Directions on how to assess performance and to qualify data.
3. Directions on how to identify trouble and to improve data quality.
4. Directions to permit design of auditing activities.

The document is not a research report. It is designed for use by operating personnel.

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by

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ABSTRACT

Guidelines for the quality control of stack gas analysis for carbon monoxide emissions by the Federal reference method (NDIR) are presented. These include:

1. Good operating practices.
2. Directions on how to assess performance and to qualify data.
3. Directions on how to identify trouble and to improve data quality.
4. Directions to permit design of auditing activities.

The document is not a research report. It is designed for use by operating personnel.

This work was submitted in partial fulfillment of contract Durham 68-02-1234 by Research Triangle Institute under the Sponsorship of the Environmental Protection Agency. Work was completed as of February 1975.

SECTION I INTRODUCTION

This document presents guidelines for developing a quality assurance program for Method 10 Determination of Carbon Monoxide Emissions from Stationary Sources. For convenience of reference, this method as published by the Environmental Protection Agency in the Federal Register, March 8, 1974, is reproduced as appendix A of this report.

This document is divided into four sections:

Section I, Introduction. The Introduction lists the overall objectives of a quality assurance program and delineates the program components necessary to accomplish the given objectives.

Section II, Operations Manual. This manual sets forth recommended operating procedures to insure the collection of data of high quality, and instructions for performing quality control checks designed to give an indication or warning that valid data or data of poor quality are being collected, allowing for corrective action to be taken before future measurements are made.

Section III, Manual for Field Team Supervisor. This manual contains directions for assessing data quality on an intrateam basis and for collecting the information necessary to detect and/or identify trouble.

Section IV, Manual for Manager of Groups of Field Teams. This manual presents information relative to the test method (a functional analysis) to identify the important operations, variables, and factors; a methodology for comparing action options for improving data quality and selecting the preferred action; and statistical properties of and procedures for carrying out a quality audit for an independent assessment of data quality.

The objectives of this quality assurance program for Method 10 are to:

1. Minimize systematic errors (biases) and to monitor and control precision within acceptable limits in the measurement process,
2. Provide routine indications for operating purposes of satisfactory performance of personnel and/or equipment,
3. Provide for prompt detection and correction of conditions that contribute to the collection of poor quality data, and

4. Collect and supply information necessary to describe the quality of the data.

To accomplish the above objectives, a quality assurance program must contain the following components:

1. Recommended operating procedures,
2. Routine training of personnel and evaluation of performance of personnel and equipment,
3. Routine monitoring of the variables and parameters, which may have a significant effect on data quality,
4. Development of statements and evidence to quality data and detect defects, and
5. Action strategies to increase the level of precision/accuracy in the reported data.

Component (2) above will be treated in the final report of this contract; all others are discussed in this report.

Implementation of a properly designed quality assurance program should enable measurement teams to achieve and maintain an acceptable level of precision and accuracy in their carbon monoxide emissions measurements. It will also allow a team to report an estimate of the precision of its measurements for each source emissions test.

Variability in emission data derived from multiple tests conducted at different times includes components caused by:

1. Process conditions,
2. Equipment and personnel variation in field procedures, and
3. Equipment and personnel variation in the laboratory.

In many instances time variations in source output may be the most significant factor in the total variability. The error resulting from this component of variation is minimized by knowing the time characteristics of the source output and sampling proportionally. The sampling period should span at least one complete output cycle when possible. If the cycle is too long, either the sample collection should be made during a portion of the cycle representative of the cycle average, or multiple samples should be collected and averaged.

Quality assurance guidelines for Method 10 as presented here are designed to insure the collection of data of acceptable quality by prevention,

detection, and quantification of equipment and personnel variations in both the field and the laboratory through:

1. Recommended operating procedures as a preventive measure,
2. Quality control checks for rapid detection of undesirable performance, and
3. A quality audit to verify independently the quality of the data.

The scope of this document has been purposely limited to that of a field and laboratory document. Additional background information will be contained in the final report under this contract.

SECTION II OPERATIONS MANUAL

2.0 GENERAL

This manual sets forth recommended procedures for determination of carbon monoxide emissions from stationary sources according to Method 10. (Method 10 is reproduced from the Federal Register, Vol. 39, No. 47, Friday, March 8, 1974, and is included as appendix A of this document.) Quality control procedures and checks designed to give an indication or warning that invalid or poor quality data are being collected are written as part of the operating procedures and are to be performed by the operator on a routine basis. In addition, the performance of special quality control procedures and/or checks as prescribed by the supervisor for assurance of data quality may be required of the operator on special occasions.

The sequence of operations to be performed for each field test is given in fig. 1. Each operation or step in the method is identified by a block. Quality checkpoints in the measurement process, for which appropriate quality control limits are assigned, are represented by blocks enclosed by double lines. Other quality checkpoints involve go/no-go checks and/or subjective judgments by the test-team members, with proper guidelines for decisionmaking spelled out in the procedures.

The precision/accuracy of data obtained from this method depends upon equipment performance and the proficiency and conscientiousness with which the operator performs his various tasks. From equipment checks through on-site measurements, calculations, and data reporting, this method is susceptible to a variety of errors. Detailed instructions are given for minimizing or controlling equipment error, and procedures are recommended to minimize operator error. Before using this document, the operator should study Method 10 as reproduced in appendix A in detail. In addition, the quality assurance documents of this series for Methods 2, 3, and 4 (refs. 1-3) should be read and followed.

To insure that all apparatus satisfies the reference method specifications, acceptance checks, as specified in section 2.1, should be performed when the apparatus is purchased and field tests must always be preceded by calibration and check-out procedures as given in sections 2.2 and 2.3 respectively. The manufacturer's recommendations should be followed when using a particular piece of equipment.

EQUIPMENT SELECTION (2.1)

1. SELECT EQUIPMENT ACCORDING TO THE GUIDELINES GIVEN IN SUBSECTION 2.1 FOR THE SOURCE TO BE TESTED.

EQUIPMENT CALIBRATION (2.2)

2. CALIBRATE EQUIPMENT ACCORDING TO SUBSECTION 2.2.

PRESAMPLING PREPARATION (2.3)

3. OBTAIN PROCESS DATA, SELECT/PREPARE SAMPLING SITE, DETERMINE LOGISTICS FOR PLACING EQUIPMENT ON-SITE, AND DETERMINE STACK CONDITIONS T_s, V_s, B_{wo} AND M_d. (SUBSECTION 2.3.1)

4. CHECK OUT SAMPLING TRAIN AND RELATED COMPONENTS. (SUBSECTION 2.3.2)

5. PACKAGE AND SHIP EQUIPMENT. (SUBSECTION 2.3.4)

ONSITE MEASUREMENTS (2.4)

6. MOVEMENT OF EQUIPMENT TO SAMPLING SITE AND SAMPLE RECOVERY AREA. (SUBSECTION 2.4.1)

7. PRELIMINARY MEASUREMENTS AND SETUP WILL INCLUDE DUCT DIMENSIONS AND PROBE POSITIONING (SUBSECTION 2.4.2)

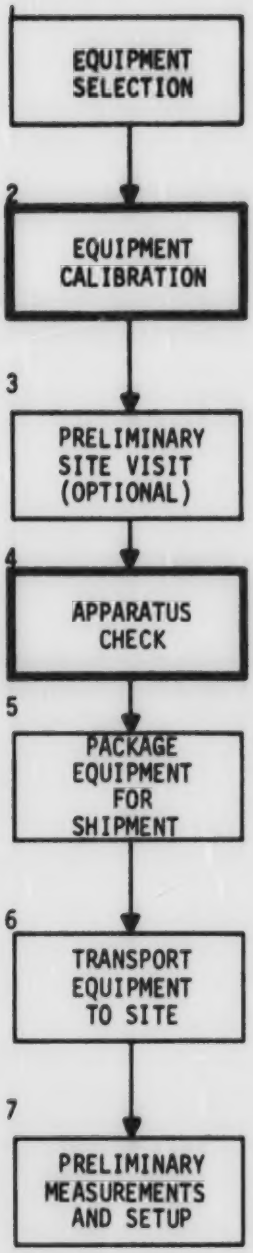


Figure 1. Operational flow chart of the measurement process.

8. DETERMINATION OF MAXIMUM AND MINIMUM ΔP AND STACK GAS TEMPERATURE. (SUBSECTION 2.4.2.2)

9. ASSEMBLE AND LEAK CHECK THE SAMPLING TRAIN. (SUBSECTION 2.4.3.2)

10. COLLECT A MINIMUM SAMPLE VOLUME OF 60 LITERS. MAINTAIN PROPORTIONAL CONDITIONS DURING SAMPLING. (SUBSECTIONS 2.4.3.3, 2.4.3.4)

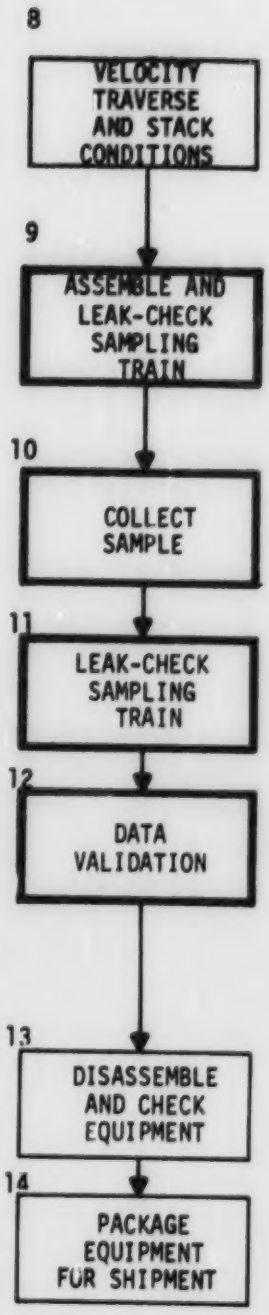
11. PERFORM FINAL LEAK CHECK OF THE ENTIRE SAMPLING TRAIN. (SUBSECTIONS 2.4.3.3, 2.4.3.4)

12. COMPARE THE NDIR MEASURED VALUES WITH THE VALUE OBTAINED BY AN ALTERNATE METHOD (3.3.3.1) OR WITH THE VALUE GIVEN BY COMBUSTION NOMOGRAPHY IF APPLICABLE. (SUBSECTION 2.5.1)

POSTSAMPLING OPERATIONS (2.5)

13. DISASSEMBLE AND INSPECT EQUIPMENT FOR DAMAGE SUSTAINED BUT NOT DETECTED DURING SAMPLING. (SUBSECTION 2.5.2)

14. PACKAGE EQUIPMENT FOR RETURN TRIP TO BASE LABORATORY. (SUBSECTION 2.5.2)



2.1 EQUIPMENT SELECTION

A listing of the required apparatus (for sampling trains configured as shown in figures 10-1 or 10-2 of appendix A) and reagents along with certain miscellaneous equipment and tools to aid in source testing is given in table 1 of subsection 2.3. Additional specifications, criteria, and/or design features as applicable are given here to aid in the selection of equipment to insure the collection of data of consistent quality. Procedures and, where applicable, limits for acceptance checks are given. The descriptive title, identification number, if applicable, and the results of the acceptance check are recorded in the receiving record file, dated, and signed by the individual performing the check. Calibration data generated in the acceptance check are recorded in a calibration log book.

2.1.1 Sampling Probe

2.1.1.1 Design Characteristics. The sampling probe should be made of stainless steel or of borosilicate (Pyrex) glass encased in a steel sheath. The probe should be equipped with a filter to remove particulate matter. This filter can be glass wool (borosilicate or quartz glass) packed in the probe end that extends into the stack. A retaining ring that screws or clamps to the end of the probe will help hold the filter in place when sampling into low-pressure stacks.

High-temperature probes (temperatures greater than 870°C) should be made of quartz. In all sampling setups, the probe material must not react with the gas constituents in a way that will introduce a bias into the analytical method.

2.1.1.2 Acceptance Check. A new probe should be visually checked for suitability, i.e., is it the length and composition ordered. The probe should be checked for cracks or breaks and leak-checked on a sampling train as described in subsection 2.2.1. Any probe not satisfying the acceptance check should be rejected.

2.1.2 Air-cooled Condenser

2.1.2.1 Design Characteristics. The purpose of the condenser is to facilitate condensation of water from the gas to be sampled. The long coiled path (figs. 10-1 and 10-2, appendix A) allows the entering gas to cool to

near ambient temperature (other temperatures can be obtained, if desired, utilizing a circulating water cooler, for example, to maintain a temperature below ambient); the large volume collects the condensed water which can be drained by the valve between or after sampling runs.

The capacity of the condenser must be sufficient to collect all condensed moisture from the sample gas during system purging and sampling, but should not be unnecessarily oversized because the added size increases the bulk of the sampling train and lengthens purge times. For example, a sample train of 1 ℓ total volume, including the condenser, should be able to hold the condensate from about 100 ℓ of sample gas (90 ℓ sample plus 5 displacements of the sampling train volume plus 5 ℓ margin). Assuming 20 percent water concentration in the stack gases, the water vapor content of this volume of stack gas is 20 ℓ of water vapor. When condensed, this gas volume corresponds to about 20 mℓ of liquid so that a condenser volume of 0.25 ℓ allows adequate operating margin.

The condenser should possess a drain valve to permit emptying between samplings.

2.1.2.2 Acceptance Check. Check the condenser visually for damage (such as breaks or cracks), and manufacturing flaws. The condenser should be airtight and leak-free when checked at a positive pressure $\geq$ 2 in. of H_2O above atmospheric and monitored with a draft gauge.

2.1.3 Needle Valves

2.1.3.1 Design Characteristics. Two metering valves with convenient-sized fittings are required in the sampling train. It is recommended that stainless steel valves be utilized.

2.1.3.2 Acceptance Check. Install the valve(s) in the sampling train and check for proper operation. Reject a valve that cannot regulate the sample flow rate over the desired operating range.

2.1.4 Vacuum Pump

2.1.4.1 Design Characteristics. The vacuum pump should be capable of maintaining: 1) a flow rate from approximately 8.5 to 34 ℓ/min (0.3 to 1.2 ft^3/min) at atmospheric pressure, and 2) creating pump inlet pressures from 25.4 to 500 mmHg with the pump outlet at or near standard pressure, i.e.,

760 mmHg. The pump must be leak-free when running and pulling a vacuum (inlet plugged) of 380 mmHg. The vacuum pump should be a leak-free diaphragm pump because of the low inherent contamination characteristics of that type of pump. For safety reasons, the pump should be equipped with a three-wire electrical cord.

2.1.4.2 Acceptance Check. A new pump should be visually checked for damage. Leak-check by plugging the pump inlet and passing the outlet line through a total volume meter such as a dry gas meter. Bubbling the outlet line through a liquid bath is an alternative method. In any event the volume of gas flowing should be less than 1 percent of the anticipated sampling rate.

2.1.5 Rate Meter

2.1.5.1 Design Characteristics. The rate meter is a rotameter or its equivalent used to measure gas flows in the range 0 to 1 ℓ/min (0 to 0.035 ft^3/min).

2.1.5.2 Acceptance Checks. Inspect the meter for cracks or flaws and check its calibration. Reject the rate meter if it is damaged, behaves erratically, or cannot be adjusted to agree within ± 5 percent of the standard rate meter. The rotameter tube and ball should be cleaned and retested if dust and/or liquid contamination is suspected.

2.1.6 Flexible Bag

2.1.6.1 Design Characteristics. The flexible bag is an inflatable, leak-tight bag used to collect a measured volume of sample gas. The bag capacity should be 60 to 90 ℓ (2 to 3 ft^3). It should be sealable to a nipple or other leak-tight connection such as illustrated in figure 10-2 of appendix A.

2.1.6.2 Acceptance Checks. Leak-test the bag in the laboratory by evacuating with a leakless pump followed by a dry gas meter. When evacuated and leak free, there should be negligible flow through the meter (less than about 10 percent bag volume in 10 hours).

One difficulty in leak-testing a nonrigid volume by evacuating it is that it is difficult to ascertain that the entire bag has been tested. What happens is that one flexible wall presses against another section and, eventually, the pumping orifice, cutting off flow. The absence of flow then does not guarantee that all sections of the bag are leak free—analogous to stopping water flow in a garden hose by pinching a section of line. This

section can be used to check for leaks between the faucet and the pinch-off point but does not test the section between the pinch-off point and the nozzel.

An alternative and preferred test method is to pressurize the bag with air to approximately 54 mm H_2O (2 in. of H_2O) above atmospheric pressure and to monitor the pressure with a draft gauge over a period of time. Any change in pressure over a 24-hour period should be considered an excessive leak and the bag should be repaired or replaced.

2.1.7 Stack Gas Velocity Temperature and Pressure Measuring System

See the quality Assurance Document of this series, entitled Determination of Stack Gas Velocity and Volumetric Flow Rate (type-S pitot tube), for a discussion of this system (ref. 1). The equipment required includes a type-S pitot tube, an inclined manometer, and appropriate lines. The pressure differential measured between the inertial pressure and the static pressure is used to adjust the sampling flow rate so that the sampling is carried out proportional to the flow rate in the stack.

2.1.8 Carbon Monoxide Analyzer

2.1.8.1 Design Characteristics. The carbon monoxide analyzer is a nondispersive infrared (NDIR) spectrometer (or its equivalent) meeting the performance specifications listed in the addenda section of appendix A.

2.1.8.2 Acceptance Checks. Demonstrate that the analyzer meets the specifications listed in appendix A as well as those advertised by the manufacturer. Guidelines for instrument evaluation are given in "Procedures for Testing Performance Characteristics of Automated Methods," Federal Register, Vol. 40, No. 33, Tuesday, February 18, 1975. A strip chart recorder is a desirable option for making a permanent record of the NDIR readings.

2.1.9 Silica Gel Drying Tube*

2.1.9.1 Design Characteristics. The drying tube is a glass tube or impinger or the equivalent capable of holding 200 grams of indicating silica gel, which removes the water vaper passing through the condenser that otherwise

*The silica gel drying tube and the CO_2 removal can be combined into one tube containing layers of the two materials. The inlet line discharges into the silica gel which grades into a mixed layer of gel and ascarite and finally becomes a layer composed totally of ascarite. The quantities of each material should be at least that specified in the standard (appendix A). The drying tube is repacked when the indicating silica gel exhibits a characteristic color change.

would interfere with the NDIR measurement of carbon monoxide. The tube should be tightly stoppered or sealed in order to be leak-free. The input and output lines should be configured to maximize the interaction between the gas and the gel. This condition is assured by adding the gel after the impinger is sealed (through the outlet line).

2.1.9.2 Acceptance Checks. Confirm that the silica gel (6 to 16 mesh) has been properly dried (2 hrs at 177° C) and indicates a dry state. Check the seal on the tube to confirm leak-free status.

2.1.10 CO_2 Removal Tube*

2.1.10.1 Design Characteristics. The CO_2 removal tube is a glass tube or impinger or the equivalent (e.g., a flexible plastic tube) capable of holding 500 g of ascarite. The dry sample gas passes through the ascarite which removes the CO_2. The tube must be leak free when loaded and sealed. The input and output lines should be configured so as to maximize the exposure of the sample gas to the ascarite. To prevent the ascarite from plugging the inlet line, the ascarite should be added through the outlet after the inlet line is sealed in place.

2.1.10.2 Acceptance Checks. Ascertain the status (remaining life) of the ascarite, replace spent ascarite. It should not be soggy or pasty. A glass wool plug on both the inlet and outlet lines helps prevent ascarite dust from being carried to other parts of the system. They should be in place or added. Confirm that the seals on the removal tube are leak free.

2.1.11.1 Design Characteristics. The ice bath must be of sufficient size to contain both the drying tube and the CO_2 removal tube in ice/ice water. While a simple bucket is suitable for onstack use, a double-walled flask with an evacuated space (a dewar flask) gives longer ice life and, hence, requires less refilling for laboratory operations.

2.1.11.2 Acceptance Check. Confirm that the bucket is large enough to hold the drying tubes surrounded by ice and water.

2.1.12 Orsat Analyzer

See the document of this series entitled, "Gas Analysis for Carbon Dioxide, Excess Air and Dry Molecular Weight," based upon method 3 (ref. 2).

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*See footnote for 2.1.9.

2.1.13 Calibration Gases

2.1.13.1 Design Characteristics. Five reference levels of carbon monoxide in nitrogen are required;

1. Zero,
2. 15 percent of span,
3. 30 percent of span,
4. 45 percent of span, and
5. 75 percent of span.

The span (reading at 100 percent of scale) should not exceed 1.5 times the applicable source standard. For example, for petroleum refineries the carbon monoxide concentration cannot exceed 500 ppm by volume (¶ 60.103 of FR 38, 15408). Instrument span, therefore, should not exceed 750 ppm. A span of 600 ppm would be reasonable with calibrating gases of 90, 180, 270, and 425 ppm concentrations in addition to the prepurified nitrogen serving as a zero calibration gas. The accuracy of the carbon monoxide concentration in the span gas determines the accuracy with which the carbon monoxide concentration in the sample gas can be measured.

2.1.13.2 Acceptance Checks. Traceability of the calibration gas to an NBS standard reference material (CO in N_2) should be established under controlled laboratory conditions prior to acceptance. A cylinder of calibration gas should not be accepted anytime the average of five or more determinations, made on different days, under controlled conditions and with an analyzer spanned with an NBS standard prior to each determination, differs from the vendor's certified value by more than $\pm$ 4 percent.

2.1.14 Tubing and Connecting Lines

2.1.14.1 Design Characteristics. Connecting lines--tubing made of the appropriate material--couple the individual components together to make up the sampling and analytical systems. For CO sampling and analysis, the range of acceptable materials is quite wide; stainless steel, aluminum, glass, plastics such as polypropylene, PVC, Teflon, and Tygon are all satisfactory. Considerations of weight, ease of assembly, or durability may favor plastic lines for many of the lengthy couplings. Short lines which are seldom touched could be made of stainless steel.

2.1.14.2 Acceptance Check. Account for all connecting lines and ascertain that they are available in the diameters, lengths, and materials desired. Make sure that all plumbing hardware is available (joints, fittings, and the tools for installation).

2.2 EQUIPMENT CALIBRATION

Before proceeding to a field site for measurements, the equipment to be used should be assembled and calibrated in a controlled laboratory setting. This section reviews calibration procedures and can be used as part of a presampling checklist for the preparation of the sampling train. In particular, new apparatus and equipment should be calibrated as part of the overall sampling system before field use, in addition to undergoing the acceptance checks for individual parts given in section 2.1.

2.2.1 Sampling Train Assembly and Checkout

Two types of sampling trains appear in Method 10: the continuous sampling train (fig. 10-1, appendix A) and the integrated gas-sampling train (fig. 10-2, appendix A).

In the continuous sampling method, the analytical equipment (fig. 10-3, appendix A) must be in position in the field to couple directly onto the sampling train. A pitot tube is not required in continuous sampling because the sampling flow rate need not be maintained proportional to the stack gas velocity. The needle valve and rate meter between the pump and the NDIR analyzer (fig. 10-3, appendix A) permit manual adjustments in accordance with the manufacturer's recommendations for gas flow and pressure during NDIR measurements.

The carbon dioxide concentration must also be determined using the integrated sampling techniques of Method 3 with either the continuous or integrated sampling techniques of Method 10*. Guidelines for this method have been previously prepared (ref. 2). The appropriate guidelines are those for the integrated sampling of CO_2 using the Orsat analyzer. As emphasized in those guidelines, using the Orsat analyzer in the stack area

*Appendix A (Method 10 Standard) describes an alternative method for determining the CO_2 concentration. In this procedure, the weight of the ascarite CO_2 removal tube before and after a given volume of sample gas has passed through it is used to calculate the average CO_2 concentration. This procedure has not worked out adequately in various round-robin checks (ref. 4) and is therefore not recommended. These guidelines assume that the Orsat method will be used.

is a last resort only. A nearby laboratory or room is far superior in regards to measurement accuracy and precision as well as analyzer maintenance and life. Similar comments apply to NDIR analytical equipment. Time-dependent permeation of CO through the flexible bag used to transport the sample is expected, so that it is important to minimize any delays between sampling and evaluation when carrying out integrated sampling.

In the integrated gas-sampling train, a sample of gas is collected over a period of time (a minimum of 60 minutes for petroleum refineries) and stored in a bag, which is subsequently transported to a convenient laboratory location for the determination of CO_2 and CO. The gas sample is taken proportionally, using a needle valve and rate meter (fig. 10-2, appendix A) to match manually changes in the sampling gas velocity to changes in the stack gas velocity (as determined by the pitot tube). When the probe position remains fixed in the vicinity of the duct centroid throughout the sampling period (no traverses of the duct), the implicit assumption is that the CO concentration is uniform across the duct. In some sampling it may be desirable to draw from different points traversing the duct.

Once collected, the sample can be transported to the analyzers, both the Orsat and the NDIR, and evaluated under controlled laboratory conditions. While in principle the orsat analyzer measures both CO_2 and CO, the minimum detectable CO level, by the Orsat method, is about 1,000 ppm--far too high for new source performance standards (NSPS) CO concentration levels. The only measurement of interest by the Orsat method, therefore, is the CO_2 concentration. The volume of sample gas required for Orsat analysis is small ($\leq 1\ell$) compared to total bag volume.

2.2.1.1 Continuous Sampling Train. The sampling probe (fig. 10-1, appendix A) and the analytical equipment (fig. 10-3, appendix A) together constitute the complete sampling train for continuous sampling. The two separately illustrated units should be directly coupled and mounted within the same enclosure for transportation and field use. The more firmly anchored and compactly housed the equipment, the better. All that has to be free is the probe, which is easily coupled to the condenser by a flexible hose made of a material inert to the sample gas such as Teflon or polyethylene.

Three checks should be carried out on the assembled sampling chain before proceeding to the field:

1. A leak test of the system,
2. A calibration of the NDIR analyzer,
3. A calibration of the Orsat analyzer.

The procedures for calibrating the analyzers are the same regardless of whether the analyzers are used with the continuous sampling train or with the integrated gas-sampling train. These procedures are therefore discussed in separate sections (2.2.3 and 2.2.4) applicable to both sampling trains. The remainder of this section describes a procedure for leak testing the continuous sampling train.

The most straightforward method for leak testing the continuous sampling train is by sealing the probe with a plug or stopper, making sure the calibrating gases are shut off, turning on the pump, and ascertaining that the gas flow rate, as indicated on the downstream rate meter, falls to zero. If the sampling train is leak free, the pump will evacuate it rapidly so that the gas flow at the downstream rate meter becomes negligibly small. The time required to evacuate depends on the pumping speed and the volume of the sampling train. The flow rate should decrease continuously to effectively zero. Leaks anywhere in the system, including the pump, establish a steady rate flow in the system which registers as a nonzero reading on the downstream rate meter. To eliminate such a condition, which would contribute a diluting effect during sampling and, hence, would introduce error into the measurements, standard leak-detection methods, such as soap-bubble painting or sequential piece-by-piece isolation, must be employed to locate and to eliminate the leak. Not until the leak rate measured on the flow meter is less than 2 percent of the planned sampling flow rate can the system be approved as acceptably leak free.

An alternative and preferred leak test procedure is to pressurize the system (i.e., the sampling train) with air to about 54 mm H_2O (2 in. of H_2O) above atmospheric pressure and monitor the pressure with a draft gauge. If there is no detectable pressure change in 10 minutes the system is considered leak-free. A system leak test should be performed before and after each field test.

2.2.1.2 Integrated Sample Train. The integrated gas-sampling train is depicted schematically in fig. 10-2 of appendix A. It is used in conjunction with the analytical equipment shown in fig. 10-3, appendix A, but has the advantage over the continuous sampling system of not having to be physically coupled to the analytical equipment. In the field, only the sampling train itself needs to be moved to the vicinity of the stack-sampling port. The analytical equipment should be at the site, however, in order to minimize the time between sampling and analysis.

Before field use the integrated sampling train should be checked for leaks. To facilitate this leak testing (but more importantly, the leak testing in the field prior to sampling), the sampling train should include connections, valves, and lines that enable the vacuum pump to perform a series of different functions without the rerouting or the making/breaking of any lines. A suitable, simple arrangement, requiring the addition of only two three-way stopcocks, is shown in fig. 2. This arrangement, while not mandatory or necessarily the best modification, will be assumed in the following discussion.

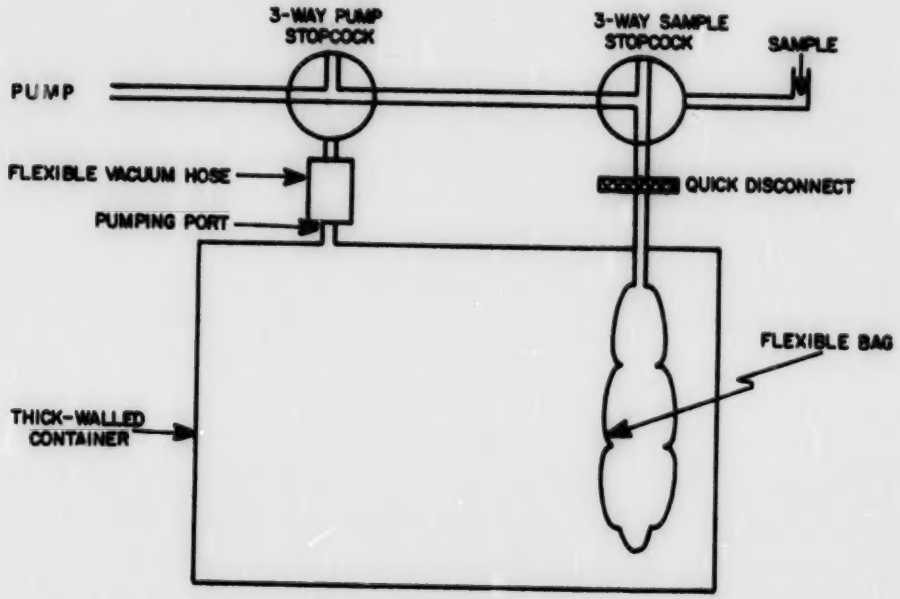


Figure 2. Modified integrated gas-sampling train.

Figure 2 illustrates the addition of two three-way stopcocks and appropriate connecting lines to the schematic appearing in the Federal Register (appendix A, fig. 10-2). These stopcocks can be made of materials compatible with the tubing used in the system. Teflon stopcocks should be adequate and relatively maintenance free. All features not shown in fig. 2 are the same as in fig. 10-2 with the exception of a rate meter which is added to the outlet line of the pump. This meter is used for leak testing. It can be of the conventional rotameter type, but a dry gas meter is preferable in terms of realiability and stability.

Once the acceptance check for the flexible bag has been completed (2.1.6.2), the bag can be attached to a finger, which is inserted through and sealed to the wall of a protective container (fig. 2). The bag is on the inside of the container; the outside end of the finger is coupled to a quick-disconnect valve. Both of these couplings are sketched in fig. 2. The bag will be used repeatedly in this configuration until it wears out, i.e., develops a leak.

The material from which the finger and container are made must be compatible with the volume requirements of the sample and with the weight restrictions of portability in the field. A heavy-walled, 20-gallon polyethylene container or drum works adequately, but other containers are suitable. The container should be relatively leak free, and while not normally subjected to large pressure differentials, it will experience large, crushing atmospheric forces. Consequently it must be rigid and strong.

Once an accepted bag has been mounted within its protective container, it remains there throughout its useful life, being filled and discharged as sampling proceeds. When it needs to be replaced, the finger is removed from the container wall, and a new bag is installed in place of the defective bag.

To check the system for leaks, the pump stopcock should be in its straight-through position, as illustrated in fig. 2. The sample stopcock can be positioned to couple the pumping line to the bag line alone, to the sample line alone, or to both together. To check the total system leak rate, the pump should be pumping on both the bag line and the sample line simultaneously (fig. 3). With the probe sealed by a stopper or other plug, the flow rate appearing on the rate meter (or the dry gas

meter) in the exhaust port of the pump is a measure of the system leak rate. This leak rate must be negligible (less than 2 percent of the anticipated sampling rate) before the sampling train is declared acceptable. The sample stopcock can be used to isolate the bag line from the sample line in tracing sources of leaks.

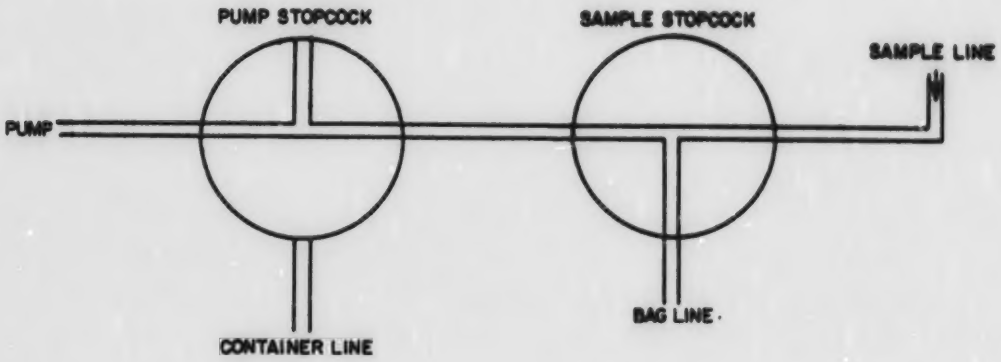


Figure 3. Stopcock configuration for determining the leak rate of the sampling train.

An alternate approach is to pressurize the bag-inlet system (2 in. of H_2O above atmospheric pressure) and monitor the change in pressure at the outlet with a draft gauge Any change in pressure over a 10 minute period is unacceptable indicating a leak in the system.

2.2.3 NDIR Analyzer Calibration

Two types of calibration procedures are given here. The first, a multipoint calibration, is a laboratory procedure designed to establish the characteristic curve relating analyzer output voltage to CO concentration flowing through the analyzer. Once prepared, this curve accompanies the analyzer and is used to convert all subsequent analyzer outputs into the parameter of interest, CO concentration.

The second calibration procedure, the zero and span calibration, is a field procedure carried out on a daily or routine basis to ascertain that the originally established calibration curve is still valid.

2.2.3.1 Multipoint Calibration Procedure. A multipoint calibration procedure is required when:

1. The analyzer is first purchased,

2. Before each set of NSPS tests and anytime the analyzer has had maintenance that could affect its response characteristics, or
3. An auditing process shows that the desired performance standards for quality of data are not being met.

A multipoint calibration requires calibrating gases with CO concentration in nitrogen, which corresponds to approximately 15, 30, 45, 75, and 100 percent of the selected instrument span, and a zero gas (the prepurified grade of nitrogen) which contains less than 0.1 mg CO/m^3.* The instrument span selected cannot exceed the applicable source performance standard by more than 1.5 times the standard. For petroleum refineries, the source standard is 500 ppm so that the maximum allowable instrument span for monitoring this industry is 750 ppm. Assuming an instrument range of 1000 ppm, the span gas then corresponds to an instrument output of about 75 percent of full scale. The remaining calibrating gases read downscale proportionally depending upon the linearity of the instrument response.

All calibration gases, certified by the vendor to be within ± 2 percent of the stated concentrations, should be purchased in high-pressure cylinders whose inside surfaces have low iron content if available. Prolonged storage in cylinders with high iron content can deplete the CO concentration through the formation of iron carbonyl. Calibration gases should be verified by establishing their traceability to an NBS standard reference material (i.e., CO in N_2) when first purchased and reverified at six month intervals. Cylinders should be stored in areas not subject to temperature extremes (i.e., not in direct sunlight).

When calibrating a specific analyzer, follow the manufacturer's detailed instructions using sound engineering judgment. General illustrative procedures are:

1. Turn the power on and let the analyzer warm up for at least 1 hour by sampling ambient air. Warm-up time varies with individual analyzers but, for field use, should not exceed several hours. The critical point is to attain temperature stability before attempting to calibrate the analyzer.
2. Connect zero gas to the analyzer.
3. Open the gas cylinder pressure valve. Adjust the secondary pressure valve to the pressure recommended by the manufacturer. Caution:

*The factor for converting CO from volume (ppm) to mass (mg/m^3) units is 1 ppm = 1.145 mg/m^3 at 25°C and 760 torr.

Do not exceed the pressure of the sample cell.

4. Set the sample flow rate as read by the rotameter (read the widest part of the float) to the value that is to be used during sampling.

5. Let the zero gas flow long enough to establish a stable trace. Allow at least 5 minutes for the analyzer to stablize. A minimum strip chart speed of 5 cm/hr (or 2 inches per hour) is recommended for this application.

6. If a recorder is used adjust the zero control knob until the trace corresponds to the line representing 5 percent of the strip-chart width above the chart zero or baseline. The above is to allow for possible negative zero drift. If the strip chart already has an elevated baseline, use it as the zero setting.

7. Let the zero gas flow long enough to establish a stable trace. Allow at least 5 minutes for this. Mark the strip chart trace as adjusted zero.

8. Disconnect the zero gas.

9. Connect the calibrating gas with a concentration corresponding to approximately 100-percent span (75-percent full scale in the case of petroleum refineries).

10. Open the gas cylinder pressure valve. Adjust the secondary pressure valve until the secondary pressure gauge reads the operations pressure recommended by the manufacturer.

11. Set the sample flow rate, as read by the rotameter, to the value that is to be used during sampling.

12. Let the span gas flow until the analyzer stabilizes.

13. Adjust the span control until the deflection corresponds to the correct percentage of chart as computed by

$$\frac{C_s(\text{ppm})}{C_f(\text{ppm})} \times 100 + 5 \text{ (percent zero offset)} = \text{correct percentage of chart}$$

where C_s = Concentration of span gas

and C_f = Full-scale reading of analyzer in same units as C_s.

As an example, see figure 4, where the percent zero offset is 5 and the correct percentage of chart for the span gas of 750 ppm would be

$$\frac{750 \text{ ppm}}{1000 \text{ ppm}} \times 100 + 5 = 80.$$

14. Allow the span gas to flow until a stable trace is observed. Allow at least 5 minutes. Mark the strip-chart trace as adjusted span and give concentration of span gas in ppm.

15. Disconnect the span gas.

16. Repeat procedures 2 through 8 and:

a. If no readjustment is required, go to Procedure 17;

b. If a readjustment greater than 2 percent of span is required, repeat procedures 9 through 16.

17. Lock the zero and span controls.

18. Connect the calibration gas with a concentration corresponding to approximately 15-percent span to the analyzer.

19. Open the gas cylinder pressure valve. Adjust the secondary pressure valve until the secondary pressure gauge reads the pressure recommended by the manufacturer.

20. Set the sample flow rate to the value used during sampling.

21. Let the calibration gas flow until the strip-chart trace stabilizes. Note: No adjustments are made at this point.

22. Disconnect the calibration gas.

23. Repeat procedures 18 through 22 for the calibration gases with concentrations corresponding to approximatelv 30, 45, and 75 percent of span.

24. Fill in the information required on a calibration sheet and construct a calibration curve of deflection as percent of chart versus concentration in ppm as illustrated in fig. 4. Draw a best-fit, smooth curve passing through the zero and span points and minimizing the deviation of the two remaining upscale points from the curve. The calibration curve should have no inflection points, i.e., it should either be a straight line or bowed in one direction only. Curve-fitting techniques may be used in constructing the calibration curve by applying appropriate constraints to force the curve through the zero and span points. This procedure becomes quite involved, however, and the most frequently used technique is to fit the curve by eye.

25. Recheck any calibration point deviating more than $\pm (9 + 0.02\ C_t)$ ppm* from the smooth calibration curve. If the recheck gives the same

*0.02 is the stated accuracy of the calibrating gas, C_t is the certified concentration of the calibrating gas and the number 9 is the 2σ limit for measuring standard samples as obtained from a collaborative study of the method (ref. 4).

Location_____Date_____Operator_____

Analyzer No._____Range_____Flow Rate_____Cell Pressure

Zero Gas_____ Cylinder Pressure_____Cylinder No._____

Upscale gas (80%)_____ Cylinder Pressure_____Cylinder No._____

(10%)_____ Cylinder Pressure_____Cylinder No._____

(20%)_____ Cylinder Pressure_____Cylinder No._____

(40%)_____ Cylinder Pressure_____Cylinder No._____

Zero Control Setting_____Span Control Setting_____

Recorder Type_____Serial No._____

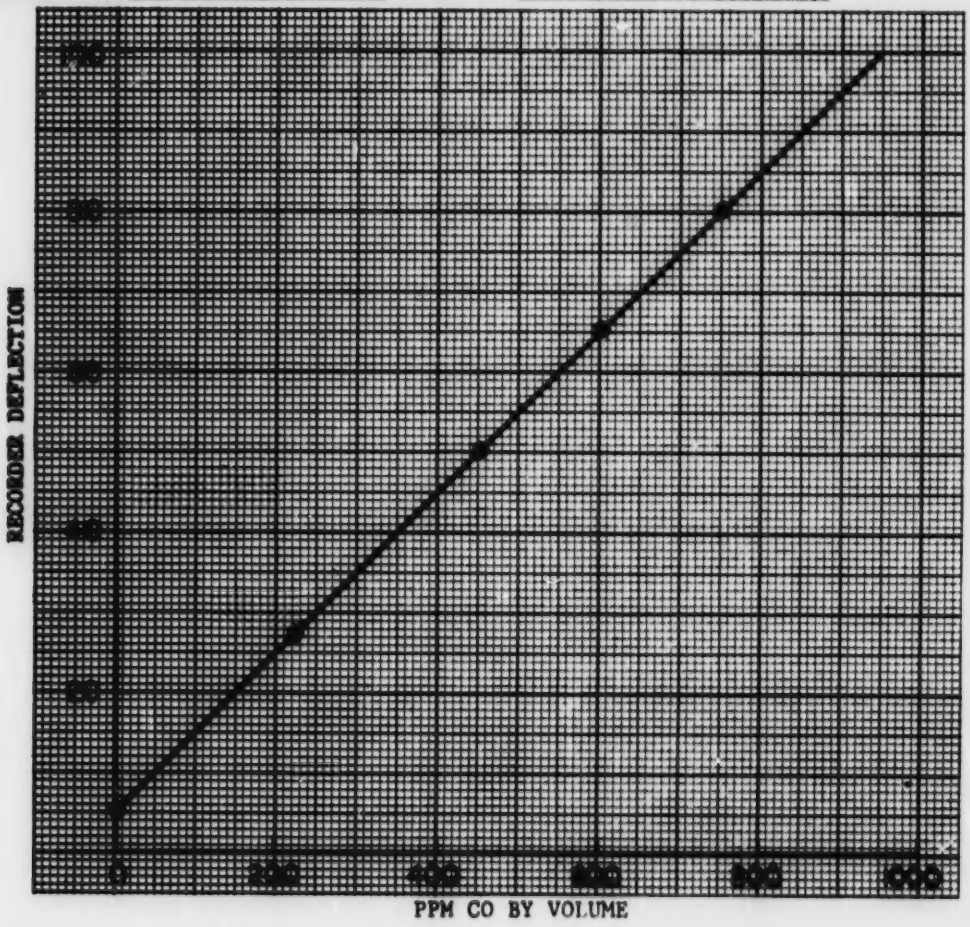


Figure 4: Sample calibration curve.

results, have that calibration gas reanalyzed. Use the best-fit curve as the calibration curve. If the calibration curve deviates more than ± 2 percent of full scale from a straight line between zero and the span point the calibration curve should be used for reducing field data.

26. In certain situations, the supervisor may request that the calibration be repeated (replicated). In this case, obtain both sets of data and follow his instructions for preparing a calibration curve.

2.2.3.2 Zero and Span Calibration

Zero and span checks are performed before and after each sample evaluation or as directed by the supervisor.

Note: In NSPS tests where only CO values close to the standard are of interest, two calibrating gases with concentrations bracketing the standard (e.g., 0.8 and 1.2 times the standard) should be used in lieu of the zero and span check as described here. Using two calibrating gases would increase the confidence in the data close to the standard and could allow for linear interpolation between the two upscale points since they are reasonably close together, for an unotherwise non-linear calibration curve.

1. Connect the calibrating gas with concentration corresponding to 100-percent span (the span gas), or other values as directed by the supervisor, to the analyzer.

2. Open the gas cylinder pressure valve and open the secondary pressure valve and let the sampler pump pull from the cylinder as needed.

3. Set the sample flow rate as read by the rotameter (read the widest part of the float) to the value to be used when sampling.

4. Let the span gas flow long enough to establish a stable trace on the strip-chart recorder; allow at least 5 minutes. Mark the chart trace as an unadjusted span. Record unadjusted span reading in ppm on the form in fig. 5, under the column entitled, "Unadjusted Calibration."

5. Disconnect the span gas.

6. Connect zero gas to the analyzer.

7. Open the gas cylinder pressure valve and adjust the secondary pressure valve until the secondary pressure gauge reads the pressure recommended by the manufacturer.

8. Set the sample flow rate as read by the rotameter to the value that is used when sampling.

CO ANALYZER DAILY CHECK SHEET

Station Name _____ Location _____

Analyzer Number _____

Date	Operator	Sample Flow Rate (ℓ/min)		Sample Cell* Pressure (Nm^{-2})		Cylinder* Pressure (Nm^{-2})		New Control Knob Setting		Unadjusted Calibration	
		Initial	Final	Initial	Final	Zero	Span	Zero	Span	Zero	Span

*newtons per square meter

Figure 5: Sample daily check sheet.

9. Let the zero gas flow long enough to establish a stable zero trace on the strip-chart recorder; allow at least 5 minutes. Mark the chart trace as an unadjusted zero. Record the unadjusted zero reading in ppm on the form in fig. 5, under the column entitled, "Unadjusted Calibration."

10. Adjust the zero control knob until the trace corresponds to the true zero setting. Let the zero gas flow until a stable trace is obtained. Mark the chart trace as an adjusted zero.

11. Disconnect the zero gas.

12. If the unadjusted zero was within $\pm$ 9 ppm of zero and the unadjusted span was within $\pm$ 9 ppm of its known value no adjustments are required and a sampling can be resumed. If either or both zero and span checks are outside the above limits continue with steps 13 through 16.

13. Reconnect the span gas and let it flow until the analyzer has stabilized; then adjust the span control until the deflection on the strip chart corresponds to the span gas concentration in ppm using the calibration curve as illustrated in fig. 4. Let the strip-chart trace stabilize. Mark the chart trace as an adjusted span with the span gas concentration in ppm.

14. Disconnect the span gas.

15. If a span adjustment greater than 2 percent of span is required, repeat procedures 6 through 13 until no adjustments are required.

16. Lock the zero and span controls.

Note: The second calibrating gas should be read at this point if bracketing the standard as described in the above note is desired.

17. Record the following information on the check sheet (see fig. 5). Record a and b under "New Control Knob Settings," and c and d under "Cylinder Pressure."

a. Zero control knob position,

b. Span control knob position,

c. Zero gas cylinder pressure (read first-stage pressure gauge),

d. Span gas cylinder pressure (read first-stage pressure gauge).

2.2.4 Orsat Analyzer Calibration

Follow the procedures given on pp. 11 and 12 of the guideline entitled, "Gas Analysis for Carbon Dioxide, Excess Air and Dry Molecular Weight" (ref. 2).

2.3 PRESAMPLING PREPARATION

2.3.1 Preliminary Site Visit (Optional)

The main purpose of a preliminary site visit is to gather information to design and implement an efficient source test. Prior preparation will result in the prevention of unwarranted loss of time, expenses, and injury to test and/or plant personnel. A test plan conceived from a thorough set of parameters will result in more precise and accurate results. This preliminary investigation (on-site) is optional and not a requirement. An experienced test group can, in most cases, obtain sufficient information about the source through communications with the plant engineer. The information should include pictures (or diagrams) of the facilities.

2.3.1.1 Process (Background Data on Process and Controls). It is recommended that the tester, before a preliminary site visit is made, become familiar with the operation of the plant. Data from similar operations that have been tested should be reviewed.

2.3.1.2 Sampling Site Preparedness. Each facility tested should provide an individual who understands the plant process and has the authority to make decisions concerning plant operation to work with the test team. This would include decisions concerning whether the plant would be operated at normal conditions or at rated capacity. This individual or individuals will supervise installation of ports, the sampling platform, and electrical power. If the above installations are already in existence, they should be examined for their suitability for obtaining a valid test and for overall safety conditions. If the sampling platform, port size, and locations are sufficient, the diameter, area of the stack, and wall thickness should be determined. If ports have to be installed, specify at least 3-inch ports (4-inch is preferred) with plugs. Port locations should be based upon Method 1 of the Federal Register (ref. 5). One electric drop with 115 volt and 20 amp service should be available at the test facility.

2.3.1.3 Stack Gas Conditions. The following can be determined on the initial site survey, either by measurement or estimation:

(1) $T_{s_{avg}}$ = Approximate stack gas temperature.

(2) P_s = The static pressure (positive or negative).

(3) ΔP_{max} and ΔP_{min} = The maximum and minimum velocity pressure heads.

(4) B_{wo} = Approximate moisture content.

(5) M_s = Molecular weight calculated from approximate gas constituent concentrations.

The above parameters can be roughly determined using an inclined manometer (0-5 inches of water), a type-S pitot tube and a manual thermometer, or thermocouple, attached to the pitot tube with a potentiometric readout device. The moisture content (approximate) can be determined by wet-bulb - dry bulb method, and the gaseous constituents by hand-held indicator kits. Nomographs are useful in checking and estimating the preliminary required data (ref. 6).

2.3.1.4 Methods and Equipment for Transporting Apparatus to Test Site. Ropes, block and tackle, and other hoisting equipment belong in the inventory of any stack sampler. The initial site visit should include the drafting of a preliminary plan by plant personnel and the test team for transporting the equipment to the sampling site. Electric forklifts should be utilized when at all possible. In addition to the above, it is recommended, when permissible, that pictures be taken of the hoisting area and sampling area, so that further discussions (either by letter or telephone) will be facilitated.

2.3.2 Apparatus Check Prior to Packing

Each item to be used should be visually checked for damage and/or excessive wear before packing. Items should be repaired or replaced as applicable, if judged to be unsuitable for use by visual inspection.

Table 1 is designed to serve as a sample checklist for the three phases of a field test. It is meant to serve as an aid to the individuals concerned with procuring and checking the required equipment and as a means for readily determining the equipment status at any point in time. The completed form should be dated, signed by the field crew supervisor, and filed in the operational log book upon completion of a field test. This includes initiating the replacement of worn or damaged items of equipment. Procedures for performing the checks are given in the appropriate subsections of this operations manual; a check is placed in the proper row and column of table 1 as the check/operation is completed. Each team will have to construct its own specific checklist according to the type of sampling train and equipment it uses.

Table 1: Apparatus Checklist for Carbon Monoxide Emissions Measurements

TEST SITE _____ CREW SUPERVISOR _____ DATE _____

ITEM	PRESAMPLING				ON-SITE MEASUREMENT			POSTSAMPLING		
	Visual Check for Damage	Leak Check Sampling Train	Performance and/or Calibration Check	Packed for Shipment	Unpacked or Purchased On-Site	Assembled and Performance Checked	Disassembled and Packed for Shipment	Inspect for Damage and/or Excess Wear	Accepted for Future Use	To Be Replaced
APPARATUS										
Sampling Train										
1. Probe										
2. Needle Valve										
3. Pump										
4. Dry Gas Meter										
5. Inclined Manometer										
6. Condenser										
7. Main Valve										
8. Flexible Bag										
9. NDIR Analyzer										
Stack Gas Velocity										
10. Type-S Pitot Tube										
11. Connecting Lines										
12. Inclined Manometer										
Stack Gas Temperature										
13. Temperature Measuring System										
Stack Gas Pressure										
14. Barometer										
15. U-Tube Manometer										
Sample Recovery										
16. Probe Brush										
17. Wash Bottles (Two)										
18. Sample Storage Containers										
19. Graduated Cylinder										
Analysis										
20. Glass Weighing Dishes										
21. Desiccator										
22. Analytical Balance										
23. Trip Balance										
REAGENTS										
Sampling										
24. Filters										
25. Silica Gel										
26. Water										
27. Crushed Ice										
28. Ascarite										
29. Calibration Gases										
Documentation										
30. Data Sheets										
Calculations										
31. Pocket Calculator (or Slide Rule)										
32. Sampling Nomograph										
Data Validation										
33. Combustion Nomographs										
TOOLS AND EQUIPMENT										
34. Equipment Transportation										
35. Safety Equipment										
36. Tools and Spare Parts										
37. Miscellaneous Supplies and Equipment										

The inclusion of spare parts, particularly those not readily available at the site, frequently pays rich dividends in reduced downtime.

2.3.3 Source Sampling Tools and Equipment

The need for specific tools and equipment will vary from test to test. A listing of the most frequently used tools and equipment is given below.

1. Transportation Equipment

a. A lightweight hand truck that can be used to transport cases and be converted to a four-wheel cart for supporting the meter box control unit.

b. A 1/2-inch, continuous-filament, nylon rope with large boat snap and snatch block for raising and lowering equipment on stacks and roofs.

c. A tarpaulin or plastic to protect equipment in case of rain. A sash cord (1/4-inch) for securing equipment and tarpaulin.

d. One canvas bucket is useful for transporting small items up and down the stack.

2. Safety Equipment

a. A safety harness with nylon and steel lanyards, large-throat snap hooks for use with lanyards for hooking over guard rails or safety line on stack.

b. A fail-safe climbing hook for use with climbing harness when climbing ladders having a safety cable.

c. Hard hats with chin straps and winter liners. Gas masks, safety glasses, and/or safety goggles.

d. Protective clothing including the following: appropriate suits for both heat and cold, gloves (both asbestos and cloth), and steeltoed shoes.

e. Steel cable (3/16-inch) with thimbles, cable clips, and turn buckles. These are required for installing a safety line or securing equipment to the stack structure.

3. Tools and Spare Parts (optional)

a. Electrical and Power Equipment

1. Circular saw
2. Variable-voltage transformer
3. Variable-speed electrical drill and bits
4. Ammeter-voltmeter-ohmeter (VOM)
5. Extension cords--light (No. 14 avg.) 2 x 25
6. Two to three wire electrical adapters
7. Three-wire electrical triple taps
8. Thermocouple extension wire
9. Thermocouple plugs
10. Fuses
11. Electrical wire

b. Tools

1. Tool boxes (one large, one small)

2. Screwdrivers
   a. one set, flat blade
   b. one set, philips
3. C-clamps (2) 6-inch, 3-inch

c. Wrenches
1. Open-end set, 1/4-inch to 1-inch
2. Adjustables (12-inch, 6-inch)
3. One chain wrench
4. One 12-inch pipe wrench
5. One Allen wrench set

d. Miscellaneous
1. Silicone sealer
2. Silicone vacuum grease (high temperature)
3. Pump oil
4. Manometers (gauge oil)
5. Antiseize compound
6. Pipe fittings
7. Dry-cell batteries
8. Flashlight
9. Valves
10. Thermometer (dial) (6-inch through 36-inch)
11. Vacuum gauge
12. SS tubing (1/4-inch, 3/8-inch, 1/2-inch) short lengths
13. Heavy-duty wire (telephone type)
14. Adjustable packing gland

2.3.4 Package Equipment for Shipment

Equipment should be packed under the assumption that it will receive severe treatment during shipment and field operation. Each item should be packaged as follows:

1. Probes, pumps, and condenser should be packed in cases or wooden boxes filled with packing material or lined with styrofoam.

2. Rotameters, needle valves, and all small, glass parts should be individually packed in a shipping container.

3. For integrated samples, it is advantageous that the rigid container for the sampling bag serve also as its shipping container.

4. The Orsat should be disassembled, and each item individually packed in suitable packing material and rigid containers. It is recommended that spare parts and the absorbent solution be shipped in another shipping container.

5. The NDIR analyzer is a self-contained instrument that should be transported in its original packing carton as initially shipped by the manufacturer or an equivalent carton to ensure the safety of the analyzer.

2.4 ON-SITE MEASUREMENTS

The on-site measurement activities include transporting the equipment to the test site, unpacking and assembling the equipment, confirming stack dimensions and traverse points (such preliminary determinations should be accomplished in a site visit), sampling, sample recovery (for integrated sampling), and data recording.

2.4.1 Transport of Equipment to the Sampling Site

The most efficient means of transporting or moving the equipment from floor level to the sampling site (as decided during the preliminary site visit) should be used to place the equipment on-site. Care should be exercised against damage to the test equipment during the moving phase. Utilization of plant personnel or equipment (winches and forklifts) under close supervison in moving the sampling gear is recommended. The CO_2 concentration determination (Orsat analysis as described in the Quality Assurance Document for Method 3 ref. 2) and the NDIR analysis of CO should be performed away from the stack or sampling area if at all possible. In most cases, the sample-recovery area can be used for gas determinations.

2.4.2 Preliminary Measurements and Setup

2.4.2.1 Stack Dimensions. Measure the stack dimensions according to subsection 2.2.3 of the Quality Assurance Document of this series for Method 2 (ref. 1). Determine the number of traverse points by Method 1 or check traverse points as determined from the preliminary site visit.

2.4.2.2 Stack Temperature and Velocity Heads. Set up and level the dual inclined manometer and determine the minimum and maximum velocity head (ΔP) and the stack temperature (T_s). This is done most efficiently with a type-S pitot tube, with a temperature-sensing device attached

The ΔP's are determined with an inclined manometer by drawing the pitot tube across the stack diameter in two directions (circular stack with 90° traverses).

2.4.3 Sampling

The on-site sampling includes preparation and assembly of the sampling train, an initial leak test, insertion of the probe into the stack, sealing the port, sampling proportionally either from the vicinity of the stack centroid or while traversing, recording of data, and a final leak test of the sampling system. Sampling is the foundation of source testing. More problems in testing result from poor or incorrect sampling than from any other part of the measurement process. The analytical process (laboratory) can never correct for errors made in the field, by either poor judgment or instrument failure. If the initial site survey, apparatus check and calibration, and preliminary measurement and setup on-site have been implemented properly, the testing should go smoothly with a minimal amount of effort and crises.

2.4.3.1 Sampling Train Assembly. Unpack and reassemble the sampling train using the identical parts that made up the train that was checked leak-free just prior to shipping. All parts should be inspected for shipping damage. Once assembled, the sampling train should again be leak-tested using the same procedures as in the preshipping checks (sec. 2.2.1.1--Continuous Sampling Train--or 2.2.1.2--Integrated Sampling Train).

After the reassembled system checks out as satisfactorily leak free, transport the system to the stack to be sampled by disconnecting only the probe from the condenser, and only if necessary; that is, the preferred technique is not to alter the system at all, but, for safe portability up the stack, a break in the system at the condenser is convenient and involves little risk to the system performance.

Field sampling most often will be with the integrated sampling system; otherwise, the NDIR analyzer must be in the vicinity of the sampling port or a long sampling line is required. Continuous sampling is used for specialized problems, such as the monitoring of short-term CO emissions--the identification of fine structure in the emission profile in time--or, in those industries, such as petroleum refineries, for which Federal regulations require continuous monitoring of CO emissions or some equivalent continuous check. Even with continuous measurement of CO, however, an

integrated sample may be used to determine the CO_2 concentration. These procedures have been described in the Guidelines for Method 3 (ref. 2).

2.4.3.2 Procedures for Continuous Sampling.

1. With the equipment in place, reconnect the probe to the condenser.

2. Repeat the leak test (sec. 2.2.1).

3. Repeat the zero and span calibration (sec. 2.2.3.2).

4. Remove the plug or cap from the probe end and confirm the position of the glass-wool filter; place the probe in the stack with the probe tip in the vicinity of the stack centroid and at least 12 inches from the stack wall.

5. Plug the sampling port to prevent dilution of the stack gas by in-leakage of ambient air. Dilution is particularly serious when the stack pressure is negative.

6. Adjust the sample gas flow rate to the desired value and purge the system by drawing a volume of sample gas through the system equal to at least five times the sampling system volume (the sampling train volume plus the volume of the gas lines in the analytical equipment).

7. Begin recording of sample CO concentration.

8. Record sample gas flow.

9. If used, check the strip-chart recorder for proper operation:

   a. Chart speed-control setting,

   b. Gain control setting,

   c. Ink trace for readability,

   d. Excess noise,

or periodically record the measured CO concentration as a function of time.

10. Adjust the flow through NDIR analyzer in accordance with manufacturer's recommendations and operational experience.

11. Upon completion of the sampling period, remove the probe and plug the open end.

12. Repeat the zero and span calibration (sec. 2.2.3.2).

13. Record the new zero and span settings or incorporate them into the strip-chart record.

14. Repeat the leak test (sec. 2.2.1).

15. Draw an integrated sample for the Method 3 determination of CO_2,

using the procedures established in the appropriate Guidelines Document (ref. 2).

Note: While the alternative method of measuring the weight increase in the ascarite over the sampling period is simpler in principle, this method has yielded unsatisfactory measurements and is therefore not recommended (ref. 4).

2.4.3.4 Procedures for Integrated Sampling.

1. With the sampling train in place on the stack, reconnect the probe to the condenser.

2. Repeat the leak test for the integrated gas sampling train (sec. 2.2.2). This procedure leaves the stopcocks positioned as in fig. 3. When the system is declared adequately leak free, the sample stopcock should be rotated 90° clockwise to isolate the sample line from the pump but not from the bag line. The stopcocks then have the configuration illustrated in figure 2.

3. Remove the plug from the end of the probe and check the position of the glass-wool filter.

4. Place the probe in the stack with the probe tip in the vicinity of the stack centroid and at least 12 inches from the stack wall.

5. Plug the sampling port with wet asbestos or other suitable material.

6. Rotate the sample stopcock 90° clockwise to couple the sample line directly to the pump and commence purging (fig. 6).

7. Purge the system by drawing a volume of sample gas equal to at least five times the volume of the sampling train.

8. Rotate the sample stopcock 90° clockwise to couple the sample line to the bag line; rotate the pump stopcock 90° counterclockwise to couple the pump to the container (fig. 7).

9. Sample at a rate proportional to the stack gas velocity as monitored by a type-S pitot tube. The rate of sampling is varied according to the variation of the square root of the velocity pressure differential, i.e., sampling rate as indicated by the rate meter is set and subsequently adjusted according to the values of $\sqrt{\Delta P}$.

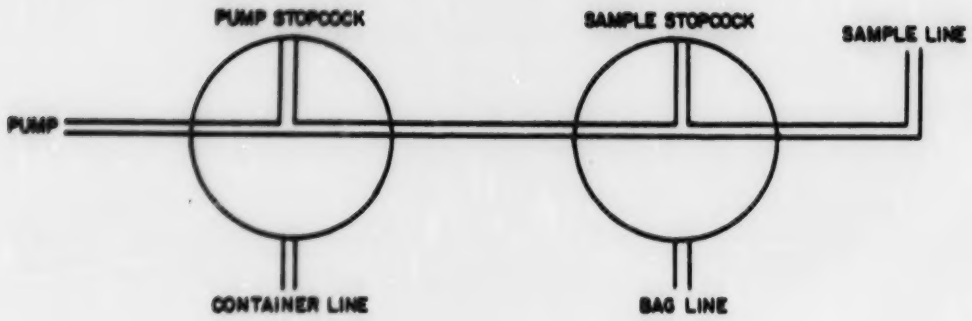


Figure 6. Stopcock configuration for purging (Integrated sampling).

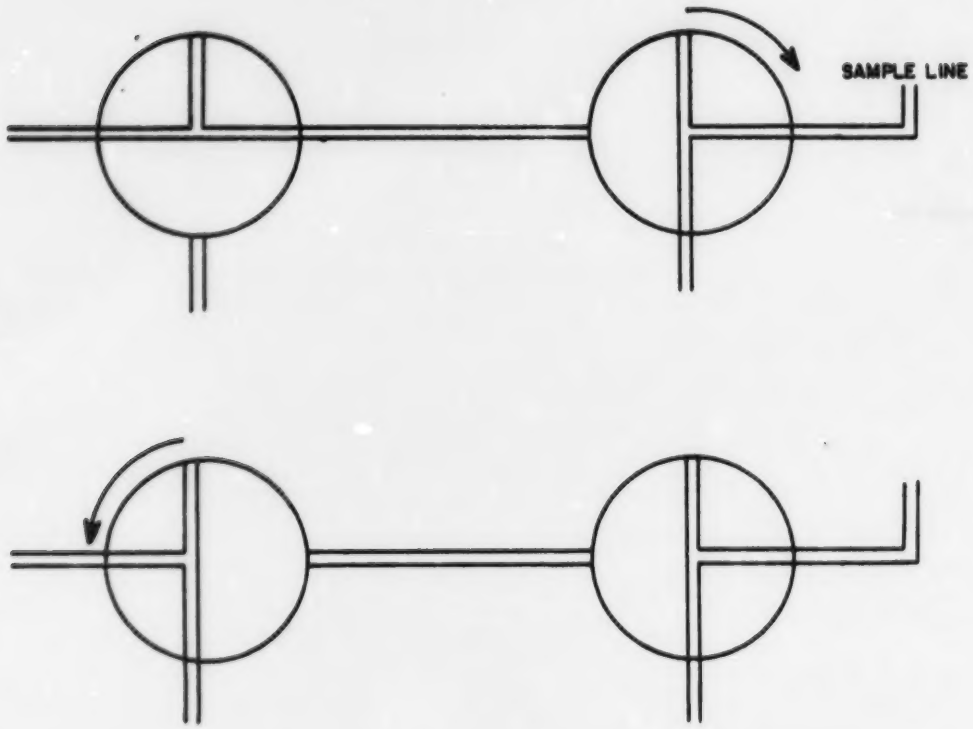


Figure 7. Stopcock movement to assume sampling configuration (Integrated sampling).

10. Disconnect the flexible sampling bag and remove it to a suitable area for performing the Orsat analysis. Use the procedures described in the Guidelines for Method 3 (ref. 2). The analysis should be performed as soon as possible but never exceeding 4 hours.

11. Record the CO_2 concentration of the sample; take at least 3 successive readings.

12. Couple the sampling bag to the NDIR analytical equipment (the analyzer should have been "on" for at least an hour).

13. Perform the zero and span calibration (sec. 2.2.3.2).

14. Purge the lines of the analyzer by drawing through them a volume of sample gas equal to at least five times the analyzer volume.

15. Record the CO concentration of the sample as determined by the NDIR analyzer take at least 3 successive readings.

16. Compute the concentration of CO in the stack using eq. 10-1 of appendix A.

2.5 POSTSAMPLING OPERATIONS

2.5.1 Data Inspection

After the analyses have been performed but before the equipment is disassembled, the measured values of CO concentration should be averaged and inspected for gross error or inconsistency. Coarse agreement should exist with the predictions of combustion nomography which estimate CO_2, CO, and O_2 concentrations when the fuel composition is known (ref. 6).

2.5.2 Equipment Disassembly, Inspection, and Packing

Once the field data have been declared acceptable as judged by the above stated points, the sampling train can be removed from the stack, dissassembled, and repacked for shipment. Defects or damage to any part of the sampling train or analytical equipment should be noted on the checklist (Table 1) for future action.

Damage that was not detected during the sampling should also be incorporated into the field data sheet. An estimate of the bias that such damage could introduce into the measurements should be made and, if significant, should be included in the field test report.

The equipment should be repacked in the same containers used to ship it to the field site. Procedures and precautions for packing are identical to those used in shipping the equipment to the field site (sec. 2.3.4).

SECTION III MANUAL FOR FIELD TEAM SUPERVISOR

3.0 GENERAL

The term "supervisor," as used in this document, applies to the individual in charge of a field team. He is directly responsible for the validity and the quality of the field data collected by his team. He may be a member of an organization that performs source sampling under contract to government or industry, a government agency performing source sampling, or an industry performing its own source sampling activities.

It is the responsibility of the supervisor to identify sources of uncertainty or error in the measurement process for specified situations and, if possible, to eliminate or minimize them by applying appropriate quality-control procedures to assure that the data collected are of acceptable quality. Specific actions and operations required of the supervisor for a viable quality-assurance program are summarized in the following list.

1. Monitor/Control Data Quality
   a) Direct the field team in performing field tests according to the procedures given in the Operations Manual.
   b) Perform or qualify results of the quality-control checks (i.e., assure that checks are valid).
   c) Perform necessary calculations and compare quality-control checks to suggested performance criteria.
   d) Make corrections or alter operations when suggested performance criteria are exceeded.
   e) Forward qualified data for additional internal review or to user.
2. Routine Operation
   a) Obtain from team members immediate reports of suspicious data or malfunctions. Initiate corrective action or, if necessary, specify special checks to determine the trouble; then take corrective action.
   b) Examine the team's log books periodically for completeness and adherence to operating procedures.
   c) Approve data sheets, data from calibration checks, etc., for filing.

3. Evaluation of Operations
   a) Evaluate available alternative(s) for accomplishing a given objective in light of experience and needs.
   b) Evaluate operator training/instructional needs for specific operations.

Consistent with the realization of the objectives of a quality assurance program as given in section I, this section provides the supervisor with brief guidelines and directions for:

1. Collection of information necessary for assessing data quality on an intrateam basis.
2. Isolation, evaluation, and monitoring of major components of system error.
3. Collection and analysis of information necessary for controlling data quality.

3.1 ASSESSMENT OF DATA QUALITY (INTRATEAM)

Intrateam or within-team assessment of data quality as discussed herein provides for an estimate of the precision of the measurements made by a particular field team utilizing an NDIR analyzer. Precision in this case refers to replicability: i.e., the variability among replicates, and is expressed as a standard deviation. This technique does not provide the information necessary for estimating measurement bias (see subsection 4.1.2 for a discussion of bias) which might occur, for example, from failure to collect a representative sample, sampling train leaks, or inadvertent exposure of the sample to ambient air. However, if the operating procedures given in the Operations Manual (section II) are followed, the bias should be small in most cases. The performance of an independent quality audit that would make possible an interteam assessment of data quality is suggested and discussed in subsection 4.2 of the Manual for Manager of Groups of Field Teams.

The field data are used to derive a confidence interval for the reported data. The primary measurement of interest here is the percent CO in the sample. Two sets of data exist as follows:

1. The Orsat determinations of CO_2 concentration;
2. the NDIR determinations of CO concentrations.

The latter (#2) can be either a continuous record corresponding to a

continuous chart trace or to a series of NDIR measurements recorded over a period of time (the continuous sampling method) or it can be the NDIR analysis of the contents of the same flexible sampling bag that was used in making the Orsat measurements of CO_2 (the integrated sampling method). Data quality checks are described for each method in the following paragraphs.

3.1.1 Continuous Sampling Method

In the continuous sampling method, the data available consist of 1) a series of CO readings, either on a strip chart or as a written, running record, including periodic measurements of zero and span drift; and 2) three Orsat measurements of the CO_2 concentration of a single integrated bag sample.

The CO_2 concentration to be used in equation 10-1 (Appendix A), which is the equation for expressing the CO concentration, is the mean of the three Orsat measurements of CO_2 concentration in the integrated bag sample. Guidelines given in the quality assurance document for Method 3 (ref. 2) should be followed in making CO_2 measurements.

Large error in the measurement of CO_2 does not have a large effect on the accuracy of the final stack CO concentration as calculated by equation 10-1 of Appendix A. Typical stack CO_2 concentrations are in the 12 percent or less range. A 50-percent error in the CO_2 measurement, therefore, produces a 6 percent or less error in the calculated CO concentration.

The continuous record of CO concentrations must be corrected for drifts in analyzer zero and span. Periodically, the continuously reading NDIR instrument should be interrupted for a zero and span calibration (2.2.3.2). For those systems using a recorder, the unadjusted zero and span should be incorporated into the strip chart, so that they can be used to correct readings for drift in both zero and span.

The procedures for correcting the chart readings for zero and span drifts are as follows:

1. Obtain the strip-chart record for the sampling period in question. The record must have adjusted span and zero traces at the beginning of the sampling period and unadjusted span and zero traces at the end of the sampling period.
2. Using a straight edge, draw a straight line from the adjusted zero at the start of the sampling period to the unadjusted zero at the

end of the sampling period. This line represents the zero baseline to be used for the sampling period.

3. Using a straight edge, draw a straight line from the adjusted span at the start of the sampling period to the unadjusted span at the end of the sampling period. This line represents the span reference line to be used for the sampling period.
4. Read the zero baseline in percent of chart at the midpoint of each hour interval (or other selected time interval).
5. Read the span reference line in percent of chart at the midpoint of each time interval as selected in step 4.
6. Determine the time interval averages by using a transparent object, such as a piece of clear plastic, with a straight edge at least 1 inch long. Place the straight edge parallel to the horizontal chart-division lines. For the interval of interest between two vertical time lines, adjust the straight edge between the lowest and highest points of the trace in that interval, keeping the straight edge parallel to the chart-division lines, until the total area above the straight edge bounded by the trace and the time lines is estimated to equal the total area below the straight edge bounded by the trace and time lines.
7. Substract the zero baseline of each interval from: a) the span reference and b) the time interval average.
8. Determine the percentage of span for each interval (the b/a ratio from step 7) and convert to ppm. by multiplying it by the span concentration.

When the data are collected by a series of recorded measurements, (i.e., no strip chart record is available), the zero and span should be checked and adjusted for each individual reading or once an hour, whichever time interval is longer. Performance specifications limit both zero and span drift to a maximum of 10 percent of full scale in 8 hours. Hourly drifts in excess of about 3 percent of full scale are symptoms of instrument instability and should be monitored closely.

3.1.2 Integrated Sampling Method

The only difference in the intrateam assessment of measurement precision using an integrated sample as opposed to continuous sampling is that the NDIR measurements of CO are made from the contents of the same flexible bags used in the Orsat determinations of CO_2.

A minimum of three readings of both CO (NDIR) and CO_2 (Orsat) concentration is made on integrated samples. The mean and standard deviation of each of these variables can be computed for three determinations as follows:

$$CO = 1/3 \sum_{i=1}^{3} CO_i, \tag{1}$$

and

$$s\{CO\} = \left[\sum_{i=1}^{3} (CO_i - CO)^2/2\right]^{1/2} \tag{2}$$

where

CO = The average of three (i=1, 2, 3) determinations, ppm

CO_i = The i^{th} CO determination after correction for CO_2 removal, ppm

$s\{CO\}$ = The standard deviation calculated from the three CO measurements, ppm.

The average of the three measurements is reported as the carbon monoxide concentration in the stack gas. The calculated standard deviation can be used to place confidence limits on the measurements.

It is recommended that the CO data be reported with 90 percent confidence limits as follows:

$$CO_t = CO \pm 2.92\ s\{CO\}/\sqrt{3} \tag{3}$$

where

CO_t = The true mean of the integrated sample

CO = The experimental mean determined from 3 measurements

2.92 = The 95th percentile of the Student t-distribution with 2 degrees of freedom which yields a 90 percent confidence interval.

$s\{CO\}/\sqrt{3}$ = The calculated standard deviation of the mean of 3 measurements.

Limits constructed in this manner will contain the true mean CO concentration

of the bag approximately 90 percent of the time, assuming that the sampling is not biased. Assessment of this assumption can be carried out by audits in which an independent inspecting team prepares reference samples with known CO, CO_2, and if desired, known levels of water vapor to be measured by the field team (sec. 4.2).

3.2 MONITORING DATA QUALITY

In general, if the procedures outlined in the operations manual are followed, the major sources of variability will be in control. It is felt, however, that as a means of verification of data quality, as well as a technique for monitoring personnel and equipment variability, two quality control charts should be constructed and maintained as part of the quality assurance program. The quality control charts will provide a basis for action with regard to the measurement process: namely, whether the measurment process is satisfactory and should be left alone, or whether the process is out of control and action should be taken to find and eliminate the causes of excess variability. In the case of this method in which documented precision data are scarce, the quality control charts can be evaluated after 20 to 30 data points have been obtained to determine the range of variation that can be expected under normal operating conditions.

The two recommended quality control charts are:

1. A range chart for the analyses performed in the field, which should serve as an effective monitor of operator variability and, to a lesser extent, of equipment variability, and
2. A chart for the differences in measured and known values (span drift), as obtained from calibration checks, to monitor equipment and/or operator variability, as well as systematic errors (biases).

Discussions of control charts and instructions for constructing and maintaining them are given in many books on statistics and quality control, such as in refs. 7 and 8.

It is good practice to note directly on control charts the reason for out-of-control conditions, if determined, and the corrective actions taken. It is also good practice to maintain control charts in large sizes, e.g., 8-1/2 x 11 inches or larger, and to keep them posted on a wall for viewing by all concerned, rather than to have them filed in a notebook (when the analyzer is housed in a laboratory type facility).

3.2.1 Range Chart

Figure 8 is a sample control chart for the range. The chart was constructed for a sample size of three; i.e., only three replicates per field test are used. It is recommended here that the range be computed for the first three analyses performed for a given field test.

For illustrative purposes, a standard deviation of 9 ppm* (see sec. 4.1.2) for the measurement error was assumed in computing $\bar{R}$ and the upper control limit (UCL). In practice the standard deviation and $\bar{R}$ computed from actual data should be used. (For small sample sizes ($r \leq 6$) the lower control limit (LCL) is effectively zero and is not given here.)

The R values are plotted sequentially by the supervisor as they are obtained from each field test and connected to the previously plotted point with a straight line. Corrective action, such as a review of operating technique should be taken any time one of the following criteria is exceeded:

1. One point falls outside the UCL.
2. Two out of three points from consecutive field tests fall in the warning zone (between 2σ and 3σ limits).
3. Points from seven consecutive field tests fall above the CL line.

Exceeding any one of the criteria will usually indicate poor technique.

3.2.2 Difference Chart

A sample quality control chart for span drifts, i.e., the difference between measured and known values of the calibration gas to be maintained on site by the field team is shown in fig. 9. The chart was constructed using a standard deviation of 9.0 ppm for the measurement error and assuming that the test gas concentrations are accurately enough known not to increase this variability substantially. It is suggested that the chart, as set up in fig. 9, be used until sufficient field data are available to compute new limits.

For each regularly scheduled span check, compute

$$d = CO_m - CO_t \qquad (4)$$

where

d = the difference in the measured and known concentration of CO, ppm

*σ_e = 13 ppm was obtained from a collaborative study of the method (ref. 4).

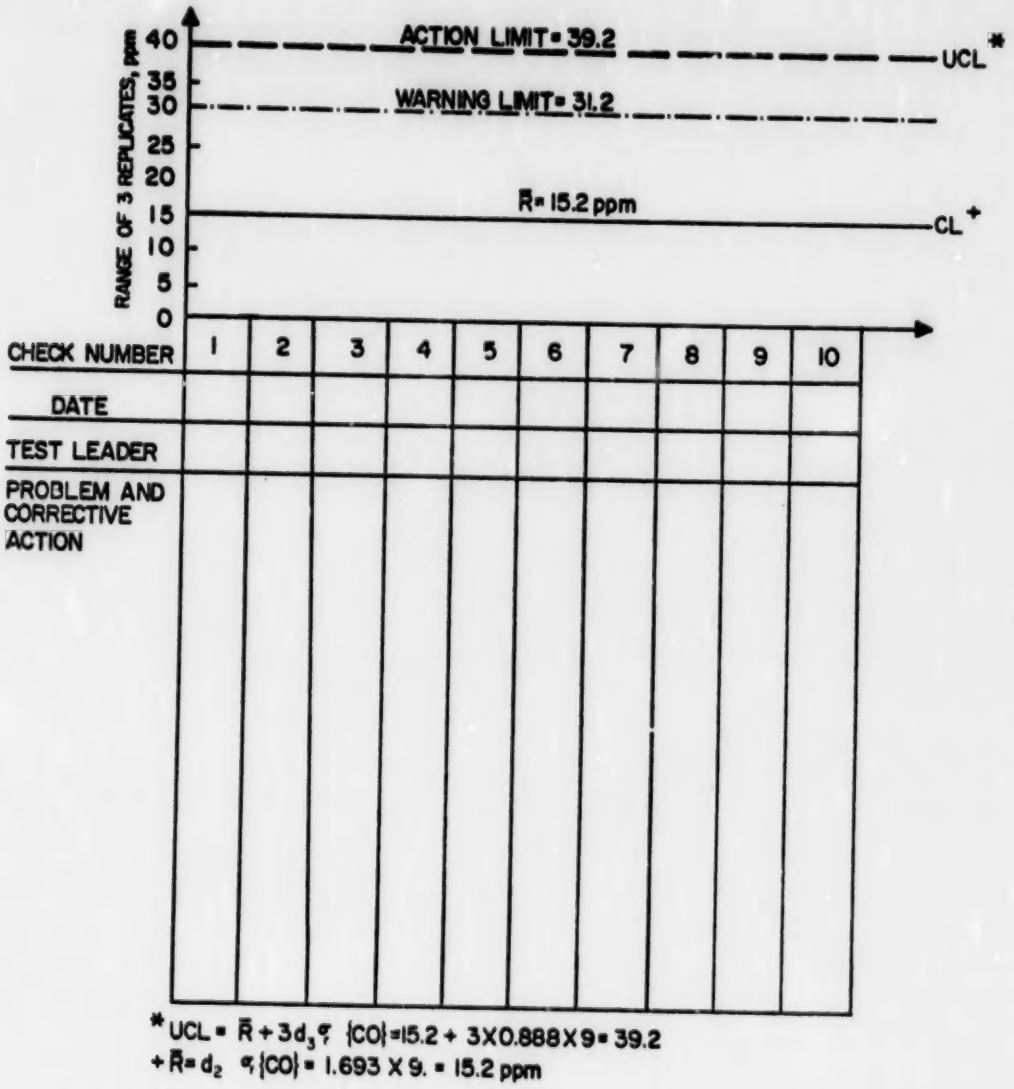


Figure 8. Sample control chart for the range, R, of field analyses.

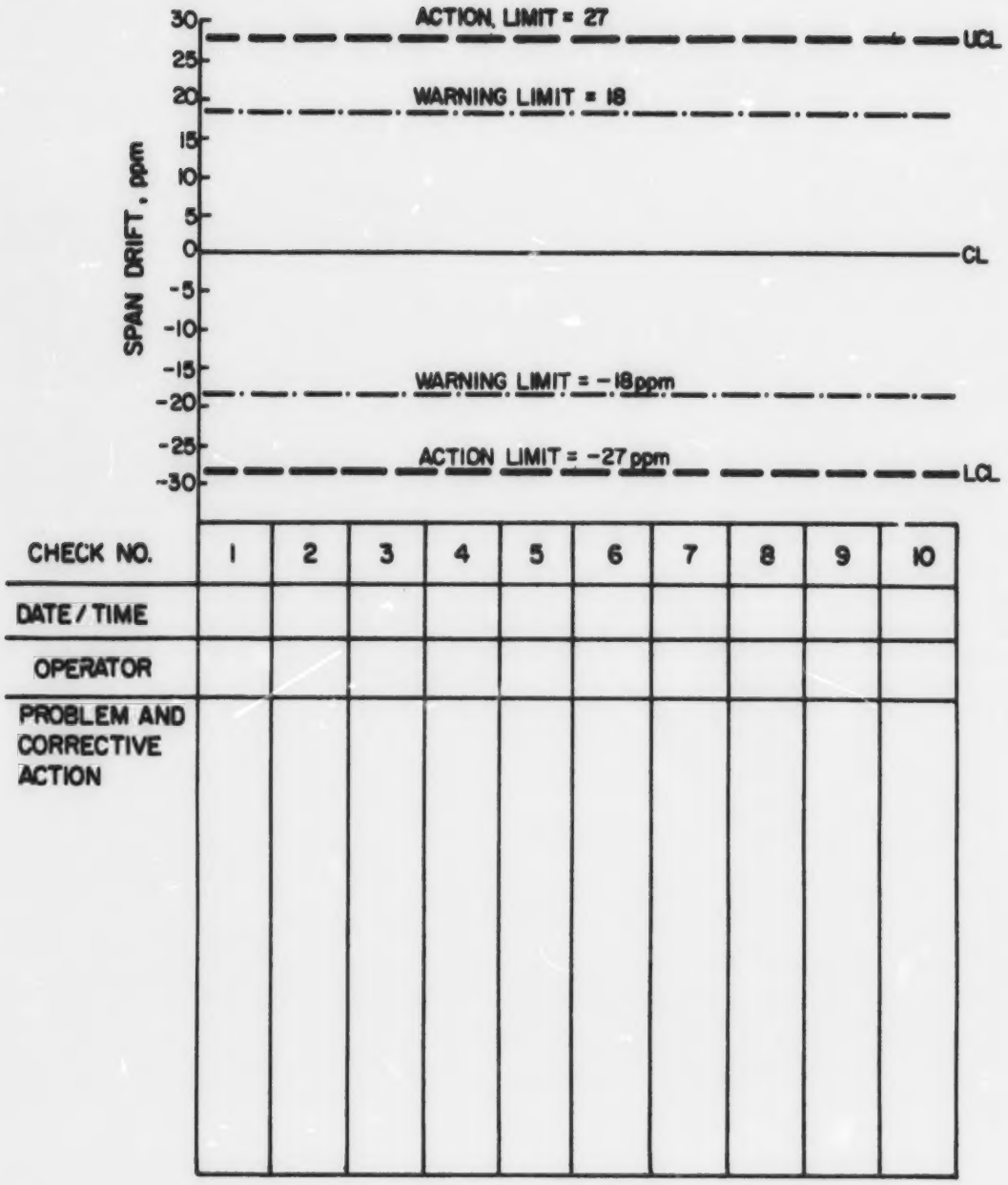


Figure 9. Sample control chart for calibration checks.

CO_m = The measured concentration of CO in ppm, and

CO_t = The true or known concentration of CO in the calibration gas in ppm.

Plot each d value on the quality control chart as it is obtained and connect it to the previously plotted point with a straight line.

Corrective action, such as recalibrating the analyzer, performing other equipment repair, and/or checking on proper operating procedures should be taken any time one of the following criteria is exceeded:

1. One point falls outside the region between the lower and upper control limits.
2. Two out of three consecutive points fall in the warning zone, i.e., between the 2σ and 3σ limits.
3. Seven consecutive points fall on the same side of the center line.

Exceeding the first or second criteria indicates excessive span drift or sudden changes in environmental conditions (e.g., temperature) or equipment malfunction and that analysis should be repeated after corrective action has been taken. The third criterion, when exceeded, indicates a system bias due to a faulty analyzer, drifting in the same direction each time, eventually resulting in the inability to properly span the analyzer.

3.3 COLLECTION AND ANALYSIS OF INFORMATION TO IDENTIFY TROUBLE

In a quality assurance program, one of the most effective means of preventing trouble is to respond immediately to indications of suspicious data or equipment malfunctions. There are certain visual and operational checks that can be performed while the measurements are being made to help assure the collection of data of good quality. These checks are written as part of the routine operating procedures in section II. In order to effectively apply preventive-type maintenance procedures to the measurement process, the supervisor must know the important variables in the process, know how to monitor the critical variables, and know how to interpret the data obtained from monitoring operations. These subjects are discussed in the following subsections.

3.3.1 Identification of Important Variables

Determination of stack gas composition requires a sequence of operations and measurements that yields, as an end result, a number that represents the average percent of a component gas for that field test. There is no way of

knowing the accuracy, i.e., the agreement between the measured and the true value, for a given field test. However, a knowledge of the important variables and their characteristics allows for the application of quality control procedures to control the effect of each variable at a given level during the field test, thus providing a certain degree of confidence in the validity of the final result.

A great many variables can affect the expected precision and accuracy of measurements made by the NDIR method. Certain of these are related to analysis uncertainties and others to instrument characteristics. Major sources of error are:

1. Inaccuracy and Imprecision in the Stated CO Concentration of Calibration Gases (ref. 9). There are two components of error involved; one is the error in the original assay, and the second is due to the deterioration of CO with time.

Large errors in the original assay should be detected when the gas is first purchased by establishing its traceability to an NBS standard reference material as described in section 2.1.13.2. Changes in concentration occurring as a function of time will be detected at a given level when reverified at six month intervals.

2. CO_2 Interferences. CO_2 is a major interference for most NDIR analyzers of CO. The technique recommended in the reference method (appendix A) is to remove the CO_2 before analysis by passing the gas stream through ascarite. The efficiency of the CO_2 removal can be variable, depending on the status of the ascarite. No compensation for variable CO_2 removal exists so that significant errors can be introduced when the ascarite loses its efficiency. If the ascarite is not new, its CO_2 removal efficiency should be checked prior to use by passing a known concentration of CO_2 (CO_2 in N_2 calibration gas) through the ascarite and observing the analyzer response. Also, such check performed after an NSPS test would ensure data free from error due to CO_2 interference.

3. Water Vapor Interference. Water vapor is a positive interference for all NDIR analyzers (ref. 9-13). The magnitude of the interference is a function of the type of water vapor control equipment being used in the measurement system and the operational state of the equipment.

Drying agents have proved to be effective in controlling water-vapor interference, but they must be checked and replaced frequently when used on

samples characterized by high relative humidities (ref. 9).

Error due to water-vapor interference is not compensated for or corrected by the zero and span calibrations. Its magnitude is monitored as part of the auditing program by the performance of periodic water-vapor interference checks.

4. Data-Processing Errors. Data processing, starting with reducing the data from a strip chart record through the act of recording the measured concentration, is subject to many types of errors. Perhaps the major source of error is in reading averages from the strip chart record. This is a subjective process and even the act of checking a given time average does not insure its absolute correctness.

The magnitude of data processing errors can be estimated from, and controlled by, the auditing program through the performance of periodic checks and making corrections when large errors are detected. A procedure for estimating the bias and standard deviation of processing errors is given in section 4.1 of the Management Manual.

5. Zero Drift. Zero drift is defined as the change in instrument output over a stated period of unadjusted, continuous operation when the input concentration is zero.

Several variables contribute to zero drift. Some variables such as variations in ambient room temperature, source voltage, and sample cell pressure result in a zero drift that is not linear with time. Therefore, performing a zero and span calibration does not correct for the component of drift throughout the sampling period but rather just at the time the calibration is performed.

Degradation of electronic components and increased accumulation of dirt in the sample cell for example may result in a zero drift that is linear with time. Periodic zero and span calibrations allow for correction of this component of zero drift for the entire sampling period.

The importance of zero drift to data quality can be determined from the results obtained from measuring control samples. For a drift that is generally linear with time, it is valid to perform zero and span calibrations before measuring control samples as part of the auditing process. However, if the drift is a function of variations in temperature, voltage, or pressure, zero and span calibrations should not be performed before measuring control samples for auditing purposes. In this case, meeting

desired performance standards may require more frequent zero and span calibrations or more rigid control of temperature, voltage, and pressure, as appropriate.

6. Span Drift. Span drift is defined as the change in instrument output over a stated time period of unadjusted, continuous operation when the input concentration is a stated upscale value. For most NDIR analyzers, the major component of span drift is zero drift and is corrected or controlled as discussed above. The component of span drift other than zero drift can be caused by either optical or electronic defects. If this component of span drift is large or shows a continuous increase with time, the manufacturer's manual should be followed for troubleshooting and correction of the defect. The importance or magnitude of span drift can be determined from the zero and span calibrations after each sampling period.

7. Excessive Noise. Noise is defined as spontaneous deviations from a mean output not caused by input concentration changes. Excessive noise may result when an analyzer is exposed to mechanical vibrations. Other sources of noise include a high gain setting on the recorder, accumulation of dirt on sample cell walls and windows, or loose dirt in the sample cell (ref. 14).

Excessive noise is evidenced by either an extra broad strip-chart trace or a narrow but erratic trace. The manufacturer's manual should be followed for troubleshooting and correcting the cause.

3.3.2 How to Monitor Important Variables

System noise, zero drift, span drift, and sample cell pressure are monitored as part of the routine operating procedures. Implementing an auditing program could effectively monitor calibration gas concentration, water vapor interference, CO_2 interference, and data-processing errors. Variations in ambient room temperature and/or source voltage can be monitored with a minimum-maximum thermometer and an a.c. voltmeter, respectively. Table 2 summarizes the variables and how they can be monitored.

3.3.3 Optional Control Procedures

Additional measurements or modified procedures can be useful in obtaining high quality data. Three recommendations are made here in response to the most common causes of measurement error: calibration gas error, ascarite failure, and bag leaks.

Table 2: Methods of monitoring variables

	Variable	Method of Monitoring
1.	Calibration Gas Concentration	Measurement of control samples as part of the auditing program.
2.	CO_2 Interference	Check for CO_2 interference by measuring calibration gases of CO_2 in N_2.
3.	Water Vapor Interference	Water vapor interference checks performed as a part of the auditing program.
4.	Data Processing Errors	Data processing checks performed as a part of the auditing program.
5.	Zero Drift	Zero check and adjustment, if required, before and after each sampling period as part of routine operating procedure.
6.	Span Drift	Span check and adjustment, if required, before and after each sampling period as part of routine operating procedure.
7.	System Noise	Check of strip chart record trace for signs of noise after each sampling period as part of routine operating procedure. When operating properly system noise should be less than 1 percent of full scale.
8.	Sample Cell Pressure Variation	Reading and recording sample cell pressure at the beginning and end of a sampling period as part of routine operating procedure
9.	Temperature Variation	Minimum-maximum thermometer placed near the analyzer, or any other temperature-indicating device, read periodically throughout the sampling period. This would usually be done as a special check. NDIR analyzers are sensitive to temperature changes. In field testing this could, if not controlled, be the greatest source of variability.
10.	Voltage Variation	A.C. voltmeter measuring the voltage to the analyzer and read periodically throughout the sampling period. This would usually be done as a special check.

3.3.3.1 Independent Back-up Determination of Bag CO Concentration. A number of alternative techniques exist for measuring CO in the concentration range of interest for stack sampling (ref. 15). Relatively inexpensive, portable instruments exist based upon such principles as:

1. The heat of combustion from the oxidation of CO in the presence of a catalyst (MSA Model D Portable CO Indicator),
2. The current flow due to an electrochemical reaction limited by the availability of CO (Ecolyzer Model 2800),
3. Solid state reactions or interactions (Bullard CO meter, CO-Dackel meter, Emmet CO meter, others).

None of these methods are as yet acceptable as equivalent standard methods, but they can, nevertheless, be very useful in identifying errors and pinpointing the source of error. Their addition to the gas analysis procedure is generally inexpensive and potentially very valuable.

What is recommended is that an auxiliary measurement of CO concentration be made on the integrated bag sample that is required for both the continuous method and the integrated method, using a measuring technique other than NDIR.

That two independent measurements of CO concentration based upon different principles can agree adds greatly to measurement confidence. If one of these two measurements is based on a chemical reaction, such as the oxidation of CO, and the other, a physical method, such as NDIR, the cross check is particularly valuable because the interferences and errors characteristic of one method are not the same as those for the other. For example, CO_2 is a major interference for the NDIR technique, and removing CO_2 is a requirement prior to the NDIR CO measurement. CO_2 is not an interference for the CO oxidation technique. And so, while both methods yield a measure of percent of CO by volume on a dry basis, the NDIR measurement relies upon efficient CO_2 removal for precision and accuracy. The chemical technique does not. General agreement in measured percent of CO by the two methods shows, among other conclusions, that the NDIR CO_2 removal technique is adequate. Conversely, agreement in the evaluation of the dry calibrating gases, but continued disagreement in the measurement of the sample gas, may mean inadequate CO_2 removal from the sample gas passing through the NDIR analyzer.

Temperature and humidity sensitivities also will be different, so that useful information can be obtained by comparing measurements. The auxiliary method will most likely be less precise than the NDIR technique. Definite standards on how well the two methods should agree cannot be given now due to insufficient data. However, at CO concentrations near 500 ppm, differences as large as 50 ppm should be taken as manifesting a need to trouble-shoot the system and reanalyze the sample.

3.3.3.2 Alternative Method for Incorporating CO_2 Correction. Ascarite failure is troublesome in that it is difficult to spot. A CO_2 in N_2 calibration gas of known concentration should be measured before and after field tests to assure and document the efficiency of the ascarite to remove CO_2.

3.3.3.3 Replacement of the Flexible Bag. The successful use of a flexible bag and the lung technique for drawing an integrated sample demands a leak-free bag. Unfortunately, the bags often leak. This defect can be indicated by a wide variation of the analyses of the three bags, drawn under essentially constant stack conditions. Leak testing the bags before each field use as recommended in section 2.1.6.2 should minimize this problem.

SECTION IV MANUAL FOR MANAGER OF GROUPS OF FIELD TEAMS

4.0 GENERAL

The guidelines for managing quality assurance programs for use with Test Method 10 - Determination of Carbon Monoxide Emissions from Stationary Sources, are given in this part of the field document. This information is written for the manager of several teams that measure source emissions and for the appropriate EPA, State, or Federal Administrators of these programs. It is emphasized that if the analyst carefully adheres to the operational procedures and checks of Section II, then the errors and/or variations in the measured values should be consistent with the performance criteria as suggested. Consequently, the auditing routines given in this section provide a means of determining whether the stack sampling test teams of several organizations, agencies, or companies are following the suggested procedures. The audit function is primarily one of independently obtaining measurements and performing calculations where this can be done. The purpose of these guidelines is to:

1. Present information relative to the test method (a functional analysis) to identify the important operations and factors.

2. Present a methodology for comparing action options for improving the data quality and selecting the preferred action.

3. Present a data quality audit procedure for use in checking adherence to test methods and for validating that performance criteria are being satisfied.

4. Present the statistical properties of the auditing procedure in order that the appropriate plan of action may be selected to yield an acceptable level of risk to be associated with the reported results.

These four purposes will be discussed in the order stated in the sections which follow. The first section will contain a functional analysis of the test method, with the objectives of identifying the most important factors that affect the quality of the reported data and of estimating the expected variation and bias in the measurements resulting from equipment and operator errors.

Section 4.2 contains several actions for improving the quality of the data; for example, by improved analysis techniques, instrumentation, and/or training programs. Each action is analyzed with respect to its potential improvement in the data quality, as measured by its precision. These results are then compared on a cost basis to indicate how to select the preferred action. The cost estimates are used to illustrate the methodology. The manager or supervisor should supply his own cost data and his own actions for consideration. If it is decided not to conduct a data audit, sections 4.1 and 4.2 would still be appropriate, as they contain a functional analysis of the reference method and of alternative methods or actions.

There are no absolute standards with which to compare the routinely derived measurements. Furthermore, the taking of completely independent measurements at the same time that the routine data are being collected (e.g., by introducing two sampling probes into the stack and collecting two samples simultaneously) although desirable is not considered practical due to the constrained environmental and space conditions under which the data are being collected. Hence, a combination of an on-site system audit, including visual observation of adherence to operating procedures and a quantitative performance quality audit check, is recommended as a dual means of independently checking on the source emissions data.

The third section contains a description of a data quality audit procedure. The most important variables identified in section 4.1 are considered in the audit. The procedure involves the random sampling of n stacks from a lot size of N = 20 stacks (or from the stacks to be tested during a 3-month period, if less than 20) for which one firm is conducting the source emissions tests. For each of the stacks selected, independent measurements will be made of the indicated variables. These measurements will be used in conjunction with the routinely collected data to estimate the quality of the data being collected by the field teams.

The data quality audit procedure is an independent check of data collection and analysis techniques with respect to the important variables. It provides a means of assessing data collected by several teams and/or firms with the potential of identifying biases/excessive variation in the data collection procedures. A quality audit should not only provide an

independent quality check, but also identify the weak points in the measurement process. Thus, the auditor, an individual chosen for his background knowledge of the measurement process, will be able to guide field teams in using improved techniques. In addition, the auditor is in a position to identify procedures employed by some field teams which are improvements over the currently suggested ones, either in terms of data quality and/or time and cost of performance. The auditor's role will thus be one of aiding the quality control function for all field teams for which he is responsible, utilizing the cross-fertilization of good measurement techniques to improve the quality of the collected and reported data.

The statistical sampling and test procedure recommended is sampling by variables. This procedure is described in section 4.4. It makes maximum use of the data collected; it is particularly adaptable to the small lot size and consequently to small sample size applications. The same sampling plans can be employed in the quality checks performed by a team or firm in its own operations. The objectives of the sampling and test procedure are to characterize data quality for the user and to identify potential sources of trouble in the data collection process for the purpose of correcting the deficiencies in data quality.

Section 4.4.4 describes how the level of auditing, sample size n, may be determined on the basis of relative cost data and prior information about the data quality. This methodology is described in further detail in the Final Report on the Contract. The costs data and prior information concerning data quality are supplied to illustrate the procedure and these data must be supplied by the manager of groups of field teams, depending upon the conditions particular to his responsibility.

Figure 10 provides an overall summary of the several aspects of the data quality assurance program as described in these documents. The flow diagram is subdivided into four areas by solid boundary lines. These areas correspond to specific sections or subsections of the document, as indicated in the upper right hand corner of each area. The details are considered in these respective sections of the document and will not be described here.

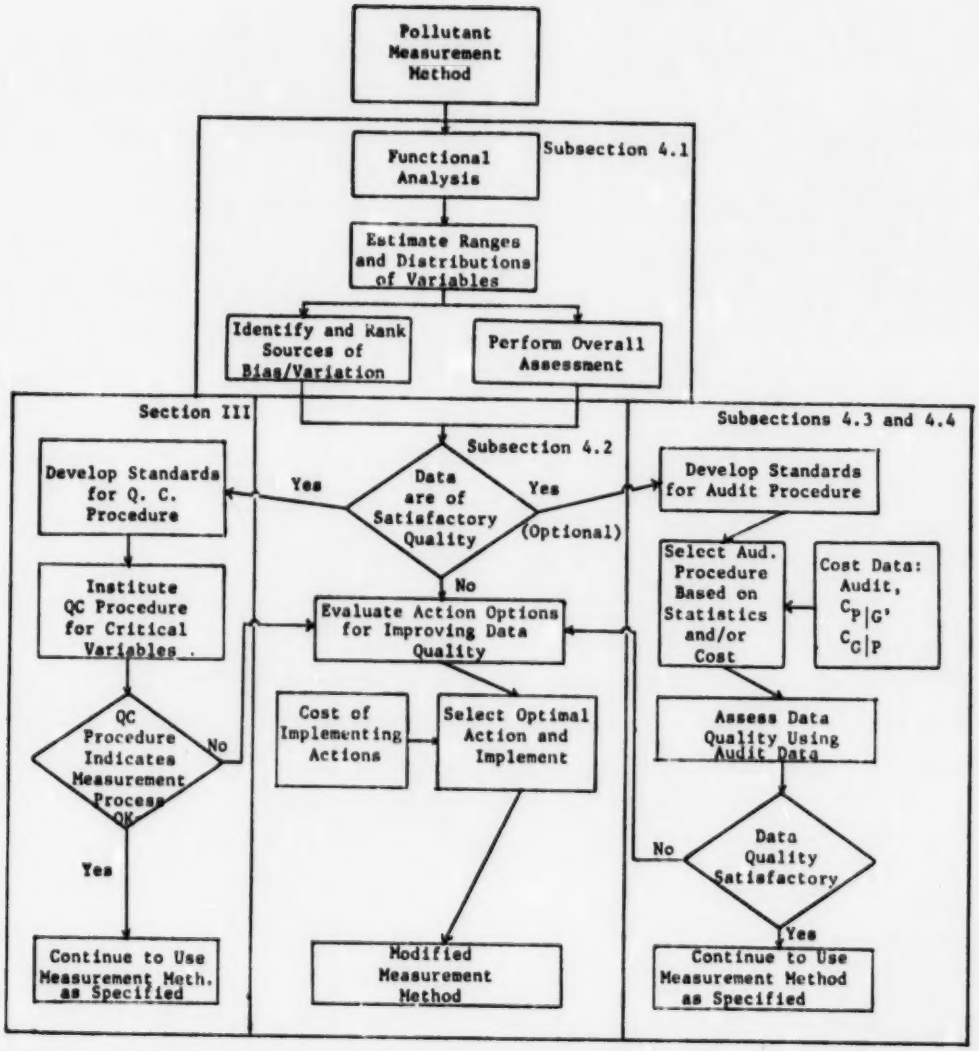


Figure 10. Summary of data quality assurance program.

4.1 FUNCTIONAL ANALYSIS OF TEST METHOD

Test method 10 - Determination of Carbon Monoxide Emission from Stationary Sources, is described in the Federal Register of March 8, 1974 and is reproduced in appendix A of this document. This method is used to determine the concentration of carbon monoxide in the stack gas on a dry basis. It requires a measurement of the CO_2 concentration in the sample, using Method 3 to make a total volume correction in the final calculation of carbon monoxide, since the CO_2 component of the sample is removed prior to NDIR analysis.

This method has not been subjected to a collaborative test; thus, quantitative information on precision and bias are not available. Therefore, the functional analysis of the method is somewhat general, using data from a collaborative test of the NDIR method of measuring CO in ambient air (ref. 9) and published data from special tests (refs. 10-14). Engineering judgements were used to estimate variable limits when data were not available. These data were used in a variance analysis to determine the resulting variability of the measured value, i.e., CO concentration.

4.1.1 Variable Evaluation and Error Range Estimates

A functional analysis for determining the CO_2 concentration by Method 3 is given in the applicable Quality Assurance Document of this series (ref. 2).

The concentration of CO in the stack gas is calculated by eq. 10-1 of appendix a, as follows:

$$CO = C_{CO_{NDIR}} (1 - F_{CO_2}) \qquad (5)$$

where

CO = Measured concentration of CO in stack on a dry basis, mg/m^3.

$C_{CO_{NDIR}}$ = Concentration of CO measured by NDIR analyzer on a dry basis, mg/m^3.

F_{CO_2} = Volume fraction of CO_2 in sample, i.e., precent CO_2 from Orsat analysis divided by 100.

The stack gas at the time and point of sample collection will have a specific but unknown CO concentration (i.e., CO^1). The difference in the

CO^1 and CO as calculated from eq. 5 above is due to a combination of errors in the measurement process. For discussion purposes the measurement process is divided into three phases. The phases are: 1) sample collection and handling, 2) sample analysis, and 3) data processing.

4.1.1.1 Sample Collection and Handling. Sample collecting and handling is subject to a variety of errors. A short description of each source of error is given in the following list.

1. Collection of the sample from one point in the stack. Collecting the sample at one point in the stack requires the assumption that the CO concentration is the same at each point in the cross-sectional plane from which the sample is taken. It also requires the assumption that the gas velocity profile remains relatively constant (i.e., if the velocity varies at one point in the plane, it varies proportionally at all points in the plane) for the sample collection period.

2. Proportional sampling. To attain a sample representative of the stack gas at the point of collection, it is necessary to maintain the sampling train flow rate proportional to the stack gas velocity. This may be difficult to do if the gas velocity changes rapidly. However, error due to deviation from proportional sampling is usually small, for gas velocity changes of less than $\pm$ 20 percent of its average velocity.

3. CO_2 interference. CO_2 is a positive interference for the NDIR method of measuring CO. Errors can result from a) incomplete removal of the CO_2, and b) an error in determining the true CO_2 concentration for making a volume correction. The potential effect on the CO measurement from either 1 or 2 above increases on an absolute basis as the CO_2 concentration increases.

4. Sampling train leaks. Leaks in the sampling train dilute the stack gas sample (both CO and CO_2) with ambient air. It is felt that the integrated gas-sampling train as shown in fig. 10-2 of appendix A is highly susceptable to leaks in the flexible bag. There are no actual data for estimating this error; however, personnel experienced in the application of this method feel that is is one of the major sources of variability.

5. H_2O interference. Water vapor is a positive interference for the NDIR method of measuring CO. Several presently unpublished tests indicate

that under normal conditions moisture contents on the order of 1 to 4 percent pass through the silica gel trap and thus to the NDIR analyzer. A rejection ratio on the order of 3.5 percent H_2O per 8 mg/m^3 (7 ppm) CO would indicate that errors as large as 8 mg/m^3 may occur frequently, and that larger errors can result if the stack gas temperature, as it leaves the silica gel trap, is not 21°C (70°F) or less and/or the silica gel is spent.

4.1.1.2 Analysis. The analysis of CO by NDIR is subject to error from inaccuracies in the calibration gases and from analyzer drifts (zero and span) due to the analyzer's sensitivity to changes in temperature and pressure.

Error in a calibration gas would bias the measurements for the lifetime of that cylinder. Analyzer drift usually is random in nature; therefore it influences the precision of the measurements. Temperature control is extremely important for NDIR analyzers. Applications in which the analyzer is exposed to the atmosphere or housed in a makeshift shelter with no temperature control will result in excess variability in the data.

4.1.1.3 Data processing. In continuous monitoring, where the CO concentration varies rapidly, an accurate estimate of the average value may be difficult to make, either from a strip chart or from discrete values recorded directly from the analyzer. Integrated sampling does not suffer from this problem since the CO concentration has reached an equilibrium in the bag.

4.1.1.4 Error range estimates. All the error terms discussed thus far are independent; at least there are no obvious reasons why they should not be independent. Therefore the total bias in the CO measurements is the sum of the biases of the individual error terms. The variance of the measurements is the sum of the variances of the individual error terms.

The variability will be larger when the measurements to be compared are performed by different analysts and/or with different equipment, than when they are carried out by a single analyst performing replicates, for example, on the same integrated sample, using the same equipment. Many different measures of variability are conceivable according to the circumstances under which the measurements are performed.

Only two extreme situations will be discussed here. They are:

1. Repeatability, r, is the value below which the absolute difference between duplicate results, i.e., two measurements made on the same sample by the same analyst using the same equipment over a short interval of time, may be expected to fall with a 95 percent probability.
2. Reproducibility, R, is the value below which the absolute difference between two measurements made on the same sample by different analysts using different equipment may be expected to fall with a 95 percent probability.

The above definitions are based on a statistical model according to which each measurement is the sum of three components:

$$CO = \overline{CO} + b + e \tag{6}$$

where

CO = the measured value, ppm

$\overline{CO}$ = the general average, ppm

b = an error representing the differences between analysts/equipment, ppm.

e = a random error occurring in each measurement, ppm.

In general, b can be considered as the sum

$$b = b_r + b_s \tag{7}$$

where b_r is a random component and b_s a systematic component. The term b is considered to be constant during any series of measurements performed under repeatability conditions, but to behave as a random variate in a series of measurements performed under reproducibility conditions. Its variance will be denoted as

$$\text{var } b = \sigma_L^2, \tag{8}$$

the between laboratory variance including the between analyst and between equipment variabilities.

The term e represents a random error occuring in each measurement. Its variance

$$\text{var } e = \sigma_r^2 \tag{9}$$

will be called the repeatability variance.

For the above model, the repeatability, r, and the reproducibility, R, are given by

$$r = 1.96 \sqrt{2}\, \sigma_r = 2.77\, \sigma_r \tag{10}$$

and

$$R = 2.77 \sqrt{\sigma_r^2/m + \sigma_L^2} = 2.77\, \sigma_R \tag{11}$$

where σ_R^2 will be referred to as the reproducibility variance and m is the number of repeated measurements averaged to give an observation, m = 1 in this case.

A collaborative study of the method (ref. 4) indicated that the reproducibility (at the 95 percent confidence level) of the method is 120 ppm i.e., R = 120 ppm and from equation 11 above σ_R = 43.5 ppm. Repeatability of the method resulting from the collaborative study is r = 97 ppm and from equation 10 σ_r = 35.5 ppm.

Bias of the method was found to vary with CO concentration and to vary between collaborators. The overall average bias derived from the measurement of standard gases was + 7 ppm.

Table 3 summarizes the previously discussed sources of error. Estimates are made of the mean, variance, and probability distribution for each variable, primarily to point out those variables that are considered to dominate the imprecision of CO measurements on repeatability and reproducibility bases. Estimates of the error involved in assuming that the CO concentration is homogeneous at all points in the sampling plane are not included in the table, due to lack of information necessary to make the estimates. All estimates are made for a true CO concentration of 500 ppm. In actual practice, precision will probably vary with concentration.

Table 3. Estimates of mean, variance, and distribution of important variables

Variable	Repeatability			Reproducibility		
	Mean ppm	Variance $(ppm)^2$	Distribution	Mean ppm	Variance $(ppm)^2$	Distribution
1. Deviation from proportional sampling*	0	100	normal	0	100	normal
2. Calibration gas	3	0	—	—	360	normal
3. CO_2 interference/volume correction	2	700	uniform	—	900	exponential
4. H_2O interference	2	64	uniform	—	100	exponential
5. Analyzer drift	0	625	normal	0	822	normal
6. Data reduction	0	87	normal	0	100	normal

*For grab sampling only

4.1.2 Variance Analysis

For the relationship

$$CO = CO_{CO_{NDIR}} (1 - F_{CO_2})$$

the variance of CO is given by

$$\sigma^2\{CO\} = (1 - F_{CO_2})^2 \sigma^2\{C_{CO_{NDIR}}\} + C^2_{CO_{NDIR}} \sigma^2\{F_{CO_2}\} \qquad (12)$$

where $\sigma^2\{C_{CO_{NDIR}}\}$ is the sum of the variances given in table 3 for either repeatability or reproducibility, whichever situation is being evaluated. From the table then $\sigma^2_r\{C_{CO_{NDIR}}\} = 1580$ and $\sigma^2_R\{C_{CO_{NDIR}}\} = 2385$. The variance $\sigma^2\{F_{CO_2}\}$ is taken from the Quality Assurance Guidelines Document for Method 3 (ref. 2) as 1.6×10^{-5}. Solving equation 12 for $C_{CO_{NDIR}} = 500$ ppm and $F_{CO_2} = 0.12$ yields

$$\sigma^2_r\{CO\} = 1225,$$

and

$$\sigma_r\{CO\} = 35 \text{ ppm},$$

or about 7 percent of the actual value.

Likewise

$$\sigma^2_R\{CO\} = 1847$$

and

$$\sigma_R\{CO\} = 43.5 \text{ ppm},$$

or about 8.7 percent of the actual value.

Repeatability, r, from equation 10 is

$$r = 2.77\ \sigma_r = 97 \text{ ppm}.$$

This says that two repeated measurements of a sample (with a CO concentration of about 500 ppm) by the same crew using the same equipment should not differ more than 97 ppm 95 percent of the time. On a relative basis, r = 19.4 percent of the actual value.

Reproducibility, R, from equation 11 gives

$$R = 120 \text{ ppm}.$$

Two measurements made on the same sample (with a CO concentration close to 500 ppm) by different teams using different equipment should agree within 120 ppm (24 percent) approximately 95 percent of the time.

4.1.3 Bias Analysis

Results of the collaborative test of the NDIR method of measuring CO in the ambient air indicated a possible positive bias of about 7 ppm. Also, in source testing it is felt that in actual field application there is a greater probability of experiencing positive interferences from H_2O and CO_2.

Assuming that the true or acceptable value, CO_t, is known from equation 6,

$$\overline{CO} - CO_t = \hat{\tau} \tag{13}$$

represents an estimate of the bias of the measurement method. In table 3, the sum of the individual variable means gives an estimated bias of

$$\hat{\tau} = 7 \text{ ppm},$$

or about +1.4 percent at a CO level of 500 ppm.

4.2 ACTION OPTIONS

Suppose it has been determined as a result of the functional analysis and/or the reported data from the checking and auditing schemes, that the data quality is not consistent with suggested standards or with the user requirements. Poor data quality may result from (1) a lack of adherence to the control procedures given in section II--Operations Manual, or (2) the need for an improved method or instrumentation for taking the measurements. It is assumed in this section that (2) applies, that is, the data quality needs to be improved beyond that attainable by following the operational procedures given for the reference method.

The selection of possible actions for improving the data quality can best be made by those familiar with the measurement process. For each action, the variance analysis can be performed to estimate the variance, standard deviation, and coefficient of variation of the pertinent measurement(s). In some cases it is difficult to estimate the reduction in

specific variances that are required to estimate the precisions of the pertinent measurements. In such cases, an experimental study should be made of the more promising actions based on preliminary estimates of precision/bias and the costs of implementing each action.

In order to illustrate the methodology, four actions and appropriate combinations thereof are suggested. Variance and cost estimates are made for each action, resulting in estimates of the overall precision of each action. The actions are as follows:

A0: Reference Method

A1: Establish traceability of calibration gas to NBS standard when first purchased (cost of $200/20 field tests)

A2: Crew training (cost of $1000/20 field tests)

A3: Special temperature control (cost of $1000/20 field tests)

A4: Check NDIR results with an alternate measurement method (cost of $250/20 field tests).

The costs given for each action are additional costs above that of the reference method. The assumptions made concerning the reduction in the variances (or improved precisions) are given in the following for each action.

1. Verification of the span calibration gas when first purchased by establishing its traceability to an NBS standard should eliminate gross errors in assayed values. It is assumed here that the mean and variance of the error due to calibration gases would be reduced from 13.5 ppm to 5 ppm and 25 to 16 respectively. Since one cylinder of span gas will last for a long time, the cost of implementing this action would be small. Also, under the assumptions made in section 4.1, this is one of the dominant sources of error,

2. From discussing this method with experienced field testers, it is felt that the method requires an operator that understands the system and its capability. Early detection of out-of-control conditions by the operator can substantially improve data quality. It is assumed here that crew training could affect all sources or variability. A reduction of σ_R from 15.5 ppm to 10 ppm and bias from 19.5 to 12 ppm is assumed as achievable with trained crews. A one week course once a year, or special OJT training, is estimated to cost approximately $1000 per 20 field tests.

3. Temperature control is critical for NDIR measurements. This implies using a laboratory or room with temperature controls to house the analyzer.

If necessary, a portable shelter should be used. An additional cost of $1000 per 20 field tests is assumed for implementation of this action. A reduction in the variance (analyzer drift) from 225 to 100 is assumed.

4. The use of an alternate measurement method to check the NDIR measurements as proposed in subsection 3.3.3.1 should aid in detecting large deviations and thus increase overall data quality. It is estimated that for a cost of about $250 per 20 field tests, the bias τ and standard deviation σ_R could be reduced to 15 ppm and 10 ppm respectively.

Figure 11 shows the results in terms of cost and data quality. Data quality for this purpose is given in terms of the Mean Square Error (MSE), which is calculated by

$$MSE = \sqrt{\tau^2 + \sigma_R^2} \qquad (14)$$

The cost of reporting poor quality data curve in figure 11, has to be derived for specific situations according to monitoring objectives. Here it

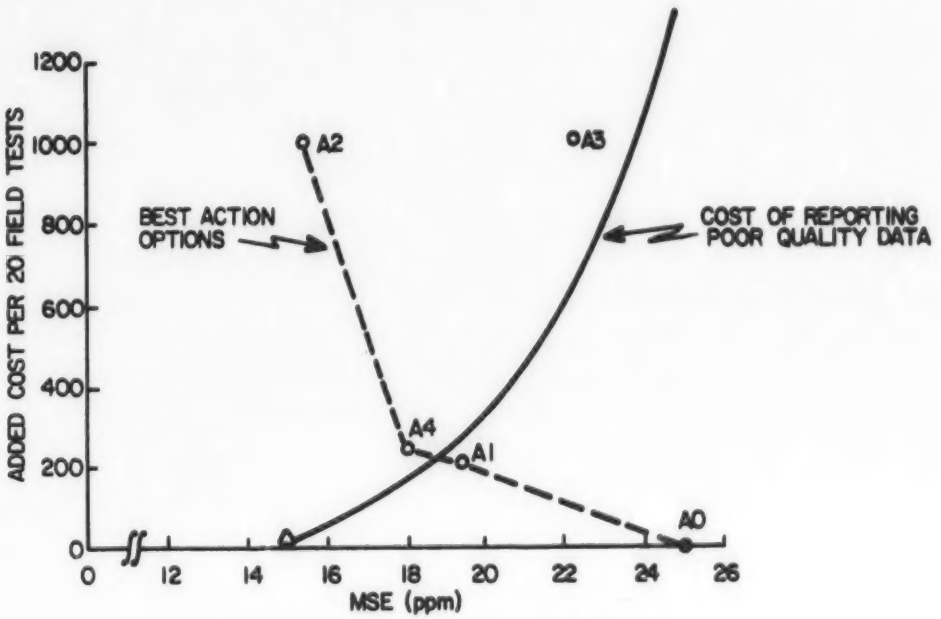


Figure 11. Added Cost versus Data Quality for Selected Action Options (at a CO level of 500 ppm).

is assumed that the cost of reporting poor quality data increases as data quality decreases. The optimum action option for this particular hypothetical situation is A_4, as seen in figure 11.

4.3 PROCEDURES FOR PERFORMING A QUALITY AUDIT

"Quality audit" as used here implies a comprehensive system of planned and periodic audits to verify compliance with all aspects of the quality assurance program. Results from the quality audit provide an independent assessment of data quality. "Independent" in this case implies that the auditor prepares a reference sample of CO and CO_2 in air and has the field team analyze the sample. The field team should not know the true CO and CO_2 concentrations. From these data both bias and precision estimates can be made for the analysis phase of the measurement process.

The auditor, i.e., the individual performing the audit, should have extensive background experience in source sampling, specifically with the characterization technique that he is auditing. He should be able to establish and maintain good rapport with field crews.

The functions of the auditor are summarized in the following list:

1. Observe procedures and techniques of the field team during on-site measurements.
2. Have field team measure sample from a reference cylinder with known CO and CO_2 concentrations.
3. Check/verify applicable records of equipment calibration checks and quality control charts in the field team's home laboratory.
4. Compare the audit value with the field team's test value.
5. Inform the field team of the comparison results specifying any area(s) that need special attention or improvement.
6. File the records and forward the comparison results with appropriate comments to the manager.

4.3.1 Frequency of Audit

The optimum frequency of audit is a function of certain costs and the desired level of confidence in the data quality assessment. A methodology for determining the optimum frequency, using relevant costs, is presented both in the Quality Assurance Documents for the methods requiring the results

of Method 3 and in the final report for this contract. Costs will vary among field teams and types of field tests. Therefore, the most cost effective auditing level will have to be derived using relevant local cost data according to the procedure given in the final report on this contract.

4.3.2 Collecting On-Site Information

While on-site, the auditor should observe the field team's overall performance of the field test. Specific operations to observe should include, but not be limited to:

1. Setting up and leak-testing the sampling train;
2. Purging the sampling train with nitrogen prior to collecting the sample;
3. Proportional sampling;
4. Frequency of zero and span checks; and
5. Transfer of sample from the flexible bag to the Orsat analyzer and the NDIR analyzer.

The above observations, plus any others that the auditor feels are important, can be used in combination to make an overall evaluation of the team's proficiency in carrying out this portion of the field test.

In addition to the above on-site observations, it is recommended that the auditor have a pressurized cylinder of CO and CO_2 in air to prepare a reference sample for analyses by the field team.

4.3.2.1 Comparing Audit and Routine Values of CO. In field tests, the audit and routine (field team's results) values are compared by

$$d_j = CO_j - CO_a \qquad (15)$$

where

d_j = The difference in the audit and field test results for the j^{th} audit, ppm

CO_a = Audit value of CO concentration, ppm

CO_j = CO concentration obtained by the field team, ppm.

Record the value of d_j in the quality audit log book.

4.3.3 Overall Evaluation of Field Team Performance

In a summary-type statement the field team should be evaluated on its overall performance. Reporting the d_j value as previously computed,

in an adequate representation of the objective information collected for the audit. However, unmeasurable errors can result from nonadherence to the prescribed operating procedures and/or from poor technique in executing the procedures. These error sources have to be estimated subjectively by the auditor. Using the notes taken in the field, the team could be rated on a scale of 1 to 5 as follows:

5 - Excellent

4 - Above average

3 - Average

2 - Acceptable, but below average

1 - Unacceptable performance.

In conjunction with the numerical rating, the auditor should include justification for the rating. This could be in the form of a list of the team's strong and weak points.

4.4 DATA QUALITY ASSESSMENT

Two aspects of data quality assessment are considered in this section. The first considers a means of estimating the precision and accuracy of the reported data, e.g., reporting the bias, if any, and the standard deviation associated with the measurements. The second consideration is that of testing the data quality against given standards, using sampling by variables. For example, lower and upper limits, L and U, may be selected to include a large percentage of the measurements. It is desired to control the percentage of measurements outside these limits to less than 10 percent. If the data quality is not consistent with the L and U limits, then action is taken to correct the possible deficiency before future field tests are performed and to correct the previous data when possible.

4.4.1 Estimating the Precision/Accuracy of the Reported Data

Methods for estimating the precision (standard deviation) and accuracy (bias) of the CO concentration were given in section 4.1. This section will indicate how the audit data collected in accordance with the procedure described in section 4.2 will be utilized in order to estimate the precision and accuracy of the measures of interest. Similar techniques can also be used by a specific firm or team to assess their own measurements. However,

in this case no bias data among firms can be obtained. The differences between the field team results and the audited results for the respective measurements are

$$d_j = CO_j - CO_{aj}. \tag{16}$$

Let the mean and standard deviation of the differences d_j; j=1, .., n be denoted by $\bar{d}$, and s_d, respectively. Thus

$$\bar{d} = \sum_{j=1}^{n} d_j/n, \tag{17}$$

and

$$s_d = \left[\sum_{j=1}^{n} (d_j - \bar{d})^2/(n-1)\right]^{1/2} \tag{18}$$

Now $\bar{d}$ is an estimate of the bias in the measurements (i.e., relative to the audited value). Assuming the audited data to be unbiased, the existence of a bias in the field data can be checked by the appropriate t-test, i.e.,

$$t = \frac{\bar{d} - 0}{s_d/\sqrt{n}}. \tag{19}$$

See ref. 16 for a discussion of the t-test.

If t is significantly large, say greater than the tabulated value of t with n - 1 degrees of freedom, which is exceeded by chance only 5 percent of the time, then the bias is considered to be real, and some check should be made for a possible cause of the bias. If t is not significantly large, then the bias should be considered zero, and the accuracy of the data is acceptable.

The standard deviation s_d is a function of both the standard deviation of the field measurements and of the audit measurements. Assuming the audit values to be much more accurate than the field measurements, then s_d is an estimate of $\hat{\sigma}\{CO\}$. Table 4, page 75 , contains an example calculation of $\bar{d}$ and s_d starting with the differences for a sample size of n = 7. (See the final report on the contract for further information concerning this result.)

The standard deviation, s_d, can be utilized to check the reasonableness of the assumptions made in section 4.1 concerning $\sigma\{CO\}$. For example, the estimated standard deviation, s_d, may be directly checked against the assumed value, $\sigma\{CO\}$, by using the statistical test procedure

$$\frac{\chi^2}{f} = \frac{s_d^2}{\sigma^2_{\{CO\}}} , \qquad (20)$$

where χ^2/f is the value of a random variable having the chi-square distribution with $f = n - 1$ degrees of freedom. If χ^2/f is larger than the tabulated value exceeded only 5 percent of the time, then it would be concluded that the test procedure is yielding more variable results due to faulty equipment or operational procedure.

The measured values should be reported along with the estimated biases, standard deviations, the number of audits, n, and the total number of field tests, N, sampled ($n \leq N$). Estimates, i.e., s_d and $\bar{d}$ which are significantly different from the assumed population parameters, should be identified on the data sheet.

The t-test and χ^2-test described above and in further detail in the final report on this contract, are used to check on the biases and standard deviations separately. In order to check on the overall data quality as measured by the percent of measurement deviations outside prescribed limits, it is necessary to use the approach described in subsection 4.4.2 below.

4.4.2 Sampling by Variables

Because the lot size (i.e., the number of field tests performed by a team or laboratory during a particular time period, normally a calendar quarter) is small, N = 20, and because the sample size is, consequently, small (of the order of n = 3 to 8), it is important to consider s sampling by variables approach to assess the data quality with respect to prescribed limits. That is, it is desirable to make as much use of the data as possible. In the variables approach, the means and standard deviations of the sample of n audits are used in making a decision concerning the data quality.

Some background concerning the assumptions and the methodology is repeated below for convenience. However, one is referred to one of a number

of publications having information on sampling by variables; e.g., see refs. 17-22. The discussion below will be given in regard to the specific problem in the variables approach, which has some unique features as compared with the usual variable sampling plans. In the following discussion, it is assumed that only CO measurements are audited as directed in sections 4.2.2.1 and 4.2.2.2. The difference between the team-measured and audited value of CO is designated as d_j, and the mean difference over n audits by $\bar{d}$ is

$$\bar{d} = 1/n \sum_{j=1}^{n} CO_j - CO_{aj}. \qquad (21)$$

Theoretically, CO and CO_a should be measures of the same CO concentration and their difference should have a mean of zero on the average. In addition, this difference should have a standard deviation equal to that associated with the measurements of CO.

Assuming three standard deviation limits, the values $\pm 3\sigma_R = \pm 131$ ppm define the respective lower and upper limits, L and U, outside of which it is desired to control the proportion of differences, d_j. Following the method given in ref. 20, a procedure for applying the variables sampling plan is described below. Figures 12 and 13 illustrate examples of satisfactory and unsatisfactory data quality with respect to the prescribed limits L and U.

The variables sampling plan requires the following information: the sample mean difference, $\bar{d}$, the standard deviation of these differences, s_d, and a constant, k, which is determined by the value of p, the proportion of the differences outside the limits of L and U. For example, if it is desired to control at 0.10 the probability of not detecting lots with data qualities p equal to 0.10 (or 10 percent of the individual differences outside L and U), and if the sample size n = 7, then the value of k can be obtained from table II of ref. 20. The values of $\bar{d}$ and s_d are computed in the usual manner; see table 4 for formulas and a specific example. Given the above information, the test procedure is applied, and subsequent action is taken in accordance with the following criteria:

Table 4. Computation of mean difference, $\bar{d}$, and standard deviation of differences, s_d

General Formulas		Specific Example	
$d = CO_j - CO_{aj}$		Data ppm	
d_1	d_1^2	-40	1600
d_2	d_2^2	+20	400
d_3	d_3^2	-10	100
d_4	d_4^2	+80	6400
d_5	d_5^2	+60	3600
d_6	d_6^2	+30	900
d_7	d_7^2	+10	100
Σd_j	Σd_j^2	+150	13,100
$\bar{d} = \frac{\Sigma d_j}{n}$		$\bar{d} = +21.4$ ppm	
$s_d^2 = \frac{\Sigma d_j^2 - \frac{(\Sigma d_j)^2}{n}}{(n-1)}$		$s_d^2 = 1650$	
$s_d = \sqrt{s_d^2}$		$s_d = 40.6$ ppm	

1. If <u>both</u> of the following conditions are satisfied,

   $\bar{d} - k\, s_d \geq L = -131$ ppm

   $d + k\, s_d \leq U = 131$ ppm

   the individual differences are considered to be consistent with the prescribed data quality limits, and no corrective action is required.
2. If one or both of these inequalities is violated, possible deficiencies exist in the measurement process as carried out for that

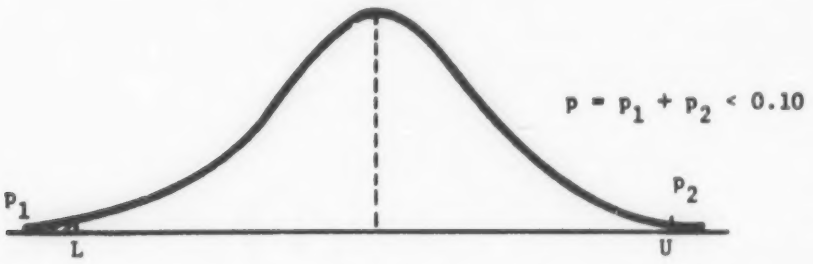


Figure 12. Example illustrating p < 0.10 and satisfactory data quality.

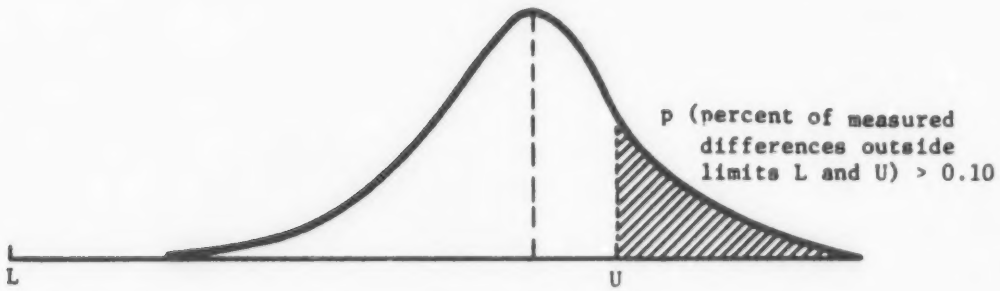


Figure 13. Example illustrating p > 0.10 and unsatisfactory data quality.

particular lot (group) of field tests. These deficiencies should be identified and corrected before future field tests are performed. Data corrections should be made when possible, i.e., if a quantitative basis is determined for correction.

Table 5 contains a few selected values of n, p, and k for convenient reference. Using the values of $\bar{d}$ and s_d in table 2, k = 2.334 for a sample size n = 7, and p = 0.10, the test criteria become

$$\bar{d} - k\, s_d = 21.4 - 2.334 \times 40.6 = -71.4 > L = -131 \text{ ppm}$$

$$\bar{d} + k\, s_d = 21.4 + 2.334 \times 40.6 = 116 \text{ ppm} < U = 131 \text{ ppm}.$$

Table 5. Sample plan constants, k for P {not detecting a lot with proportion p outside limits L and U} $\leq$ 0.1

Sample Size n	p = 0.2	p = 0.1
3	3.039	4.258
5	1.976	2.742
7	1.721	2.334
10	1.595	2.112
12	1.550	2.045

Therefore, both conditions are satisfied and the lot of N = 20 measurements is consistent with the prescribed quality limits. The plan is designed to aid in detecting lots with 10 percent or more defects (deviations falling outside the designated limits L and U) with a risk of 0.10; that is, on the average, 90 percent of the lots with 10 percent or more defects will be detected by this sampling plan.

4.4.4 Cost Versus Audit Level

The determination of the audit level (sample size n) to be used in assessing the data quality, with reference to prescribed limits L and U, can be made either 1) on a statistical basis, by defining acceptable risks for type I and type II errors, knowing or estimating the quality of the incoming data, and specifying the described level of confidence in the reported data, or 2) on a cost basis, as described herein. In this section, cost data associated with the audit procedure are estimated or assumed, for the purpose of illustrating a method of approach and identifying which costs should be considered.

A model of the audit process, associated costs, and assumptions made in the determination of the audit level is provided in figure 14. It is assumed that a collection of source emissions tests for N stacks is to be made by a particular firm, and that n measurements ($n \leq N$) are to be audited at a cost, $C_A = b + cn$, where b is a constant independent for n and c is the cost per stack measurement audited. In order to make a specific determination of n, it is also necessary to make some assumptions about the

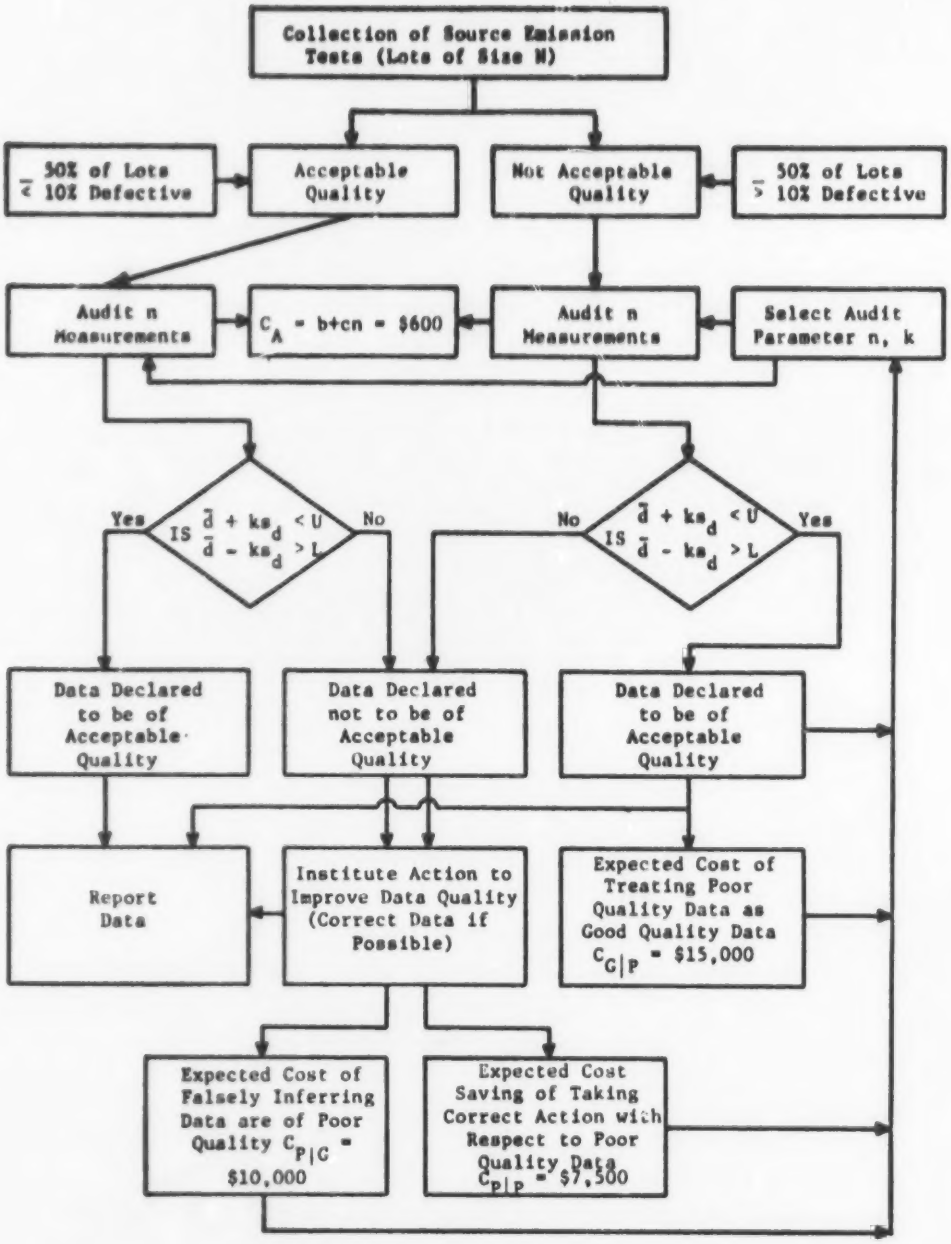

Collection of Source Emission Tests (Lots of Size N)
≤ 50% of Lots ≤ 10% Defective
Acceptable Quality
Not Acceptable Quality
≥ 50% of Lots ≥ 10% Defective
Audit n Measurements
C_A = b+cn = $600
Audit n Measurements
Select Audit Parameter n, k
Yes
IS $\bar{d} + ks_d < U$ $\bar{d} - ks_d > L$
No
No
IS $\bar{d} + ks_d < U$ $\bar{d} - ks_d > L$
Yes
Data Declared to be of Acceptable Quality
Data Declared not to be of Acceptable Quality
Data Declared to be of Acceptable Quality
Report Data
Institute Action to Improve Data Quality (Correct Data if Possible)
Expected Cost of Treating Poor Quality Data as Good Quality Data $C_{G|P}$ = $15,000
Expected Cost of Falsely Inferring Data are of Poor Quality $C_{P|C}$ = $10,000
Expected Cost Saving of Taking Correct Action with Respect to Poor Quality Data $C_{P|P}$ = $7,500

Figure 14. Flow chart of the audit level selection process.

quality of the source emissions data from several firms. For example, it is assumed in this analysis that 50 percent of the data lots are of good quality, i.e., one-half of the firms are adhering to good data quality assurance practice, and that 50 percent of the data lots are of poor quality. Based on the analysis in section 4.1, good quality data is defined as that which is consistent with the estimated precision/bias using the reference method. Thus if the data quality limits L and U are taken to be the lower and upper 3σ limits, corresponding to limits used in a control chart, the quality of data provided by firmly adhering to the recommended quality assurance procedures should contain at most about 0.3 percent defective measurements (i.e., outside the limits defined by L and U). Herein, good quality data is defined as that containing at most 10 percent defective measurements. The definition of poor quality data is somewhat arbitrary; for this illustration it is taken as 25 percent outside L and U.

In this audit procedure, the data are declared to be of acceptable quality if both of the following inequalities are satisfied:

$$\bar{d} + ks_d < U$$

$$\bar{d} - ks_d > L \ ,$$

where $\bar{d}$ and s_d are the mean and standard deviation of the data quality characteristic (i.e., the difference of the field and audited measurements) being checked. The data are not of desired quality if one or both inequalities is violated, as described in section 4.3. The costs associated with these actions are assumed to be as follows:

C_A = Audit cost = b + cn. It is assumed that b is zero for this example, and c is taken as \$600/measurement.

$C_{P|G}$ = Cost of falsely inferring that the data are of poor quality, P, given that the data are of good quality, G. This cost is assumed to be one-half the cost of collecting emissions data for N = 20 stacks (i.e., 0.5 x \$1000 x 20 = \$10,000). It would include the costs of searching for an assignable cause of the inferred data deficiency when none exists, of partial repetition of data collection, and of decisions resulting in the purchase of equipment to reduce emission levels of specific pollutants, etc.

$C_{G|P}$ = Cost of falsely stating that the data are of good quality, G, given that they are of poor quality, P. This cost is assumed to be \$15,000 (= 0.75 x \$1000 x 20), and is associated with health effects, litigation, etc.

$C_{P|P}$ = Cost savings resulting from correct identification of poor quality data. This cost is taken to be \$7,500, i.e., equal to one-half of $C_{P|G}$ or equal to 0.375 x \$1,000 x 20, the total cost of data collection.

These costs are given in figure 15. The cost data are then used in conjunction with the a priori information concerning the data quality, to select an audit level n. Actually, the audit procedure requires the selection of the limits L and U, n, and k. L and U are determined on the basis of the analysis of section 4.1. The value of k is taken to be the value associated with n in table 5 of section 4.4.3, i.e., the value selected on a statistical basis to control the percentage of data outside the limits L and U. Thus, it is only necessary to vary n and determine the corresponding expected total cost E(TC) using the following cost model

$$E(TC) = - C_A - 0.5\, P_{P|G}\, C_{P|G} + 0.5\, P_{P|P}\, C_{P|P} - 0.5\, P_{G|P}\, C_{G|P} \qquad (22)$$

where the costs are as previously defined. The probabilities are defined in a way similar to defining corresponding costs:

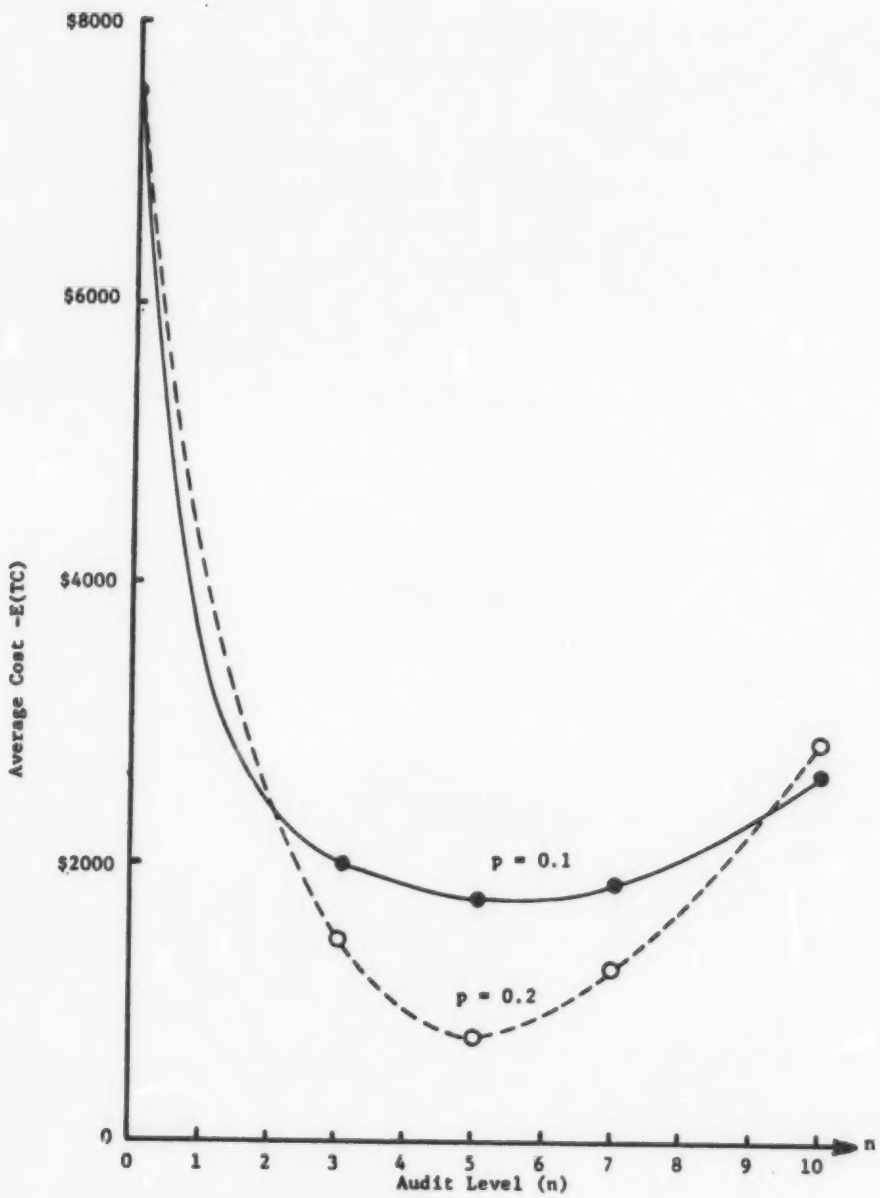
$P_{P|G}$ = Probability that a lot of good quality data is falsely inferred to be of poor quality, due to the random variations in the sample mean $\bar{d}$ and standard deviation, s_d, in small samples of size n.

$P_{P|P}$ = Probability that a lot of poor quality data is correctly identified as being of poor quality.

$P_{G|P}$ = Probability that a lot of poor quality data is incorrectly judged to be of good quality, due to sampling variations of $\bar{d}$ and s.

These three probabilities are conditional on the presumed lot quality and are preceded by a factor of 0.5 in the total cost model, to correspond to the assumed percentage of good (poor) quality data lots.

In order to complete the determination of n, it is necessary to calculate each of the conditional probabilities, using the assumptions stated



[p = Proportion defective measurements in the "lot"
P{Acc. lot with p} ≤ 0.1]

Figure 15. Average Cost vs Audit Level (n)

for a series of values of n (and associated k, which is given in table 5). The computational procedure is given in the Final Report of this contract. These calculations were made for the cases n = 3, 5, 7, and 10 and for two degrees of control on the quality of the data that can be tolerated, i.e., p = 0.2 and p = 0.1, the portion outside the limits L and U for which it is desired to accept the data as good quality, with probability less than or equal to 0.10. These computed probabilities are then used in conjunction with the costs associated with each condition, applying equation (22) to obtain the average cost versus sample size n for the two cases p = 0.1 and 0.2. The curves obtained from these results are given in figure 15. It can be seen from these curves that the minimum cost is obtained by using n = 5 independent of p. However, it must be recognized that the costs used in the example are for illustrative purposes and may vary from one region to another; thus, within the reasonably uncertainty of the estimated costs, suggest that p = 0.2 is more cost effective; this tends to permit data of poorer quality to be accepted.

SECTION V

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APPENDIX A REFERENCE METHOD FOR DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

METHOD 10—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability.*

1.1 *Principle.* An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 *Applicability.* This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

2. *Range and sensitivity.*

2.1 *Range.* 0 to 1,000 ppm.

2.2 *Sensitivity.* Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences.* Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water (H_2O) and carbon dioxide (CO_2) are 3.5 percent H_2O per 7 ppm CO and 10 percent CO_2 per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent H_2O per 35 ppm CO and 10 percent CO_2 per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

4. *Precision and accuracy.*

4.1 *Precision.* The precision of most NDIR analyzers is approximately ±2 percent of span.

4.2 *Accuracy.* The accuracy of most NDIR analyzers is approximately ±5 percent of span after calibration.

5. *Apparatus.*

5.1 *Continuous sample* (Figure 10-1).

5.1.1 *Probe.* Stainless steel or sheathed Pyrex[1] glass, equipped with a filter to remove particulate matter.

5.1.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2 *Integrated sample* (Figure 10-2).

5.2.1 *Probe.* Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2.3 *Valve.* Needle valve, or equivalent, to to adjust flow rate.

5.2.4 *Pump.* Leak-free diaphragm type, or equivalent, to transport gas.

5.2.5 *Rate meter.* Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min. (0.035 cfm).

5.2.6 *Flexible bag.* Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft^3). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

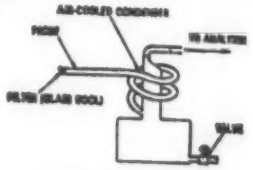

Figure 10-1. Continuous sampling train.

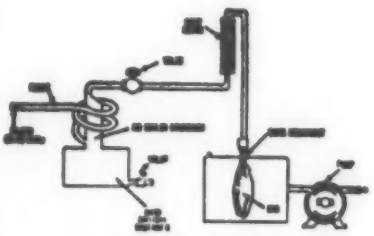

Figure 10-2. Integrated gas-sampling train.

5.2.7 *Pitot tube.* Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 *Analysis* (Figure 10-3).

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

5.3.1 *Carbon monoxide analyzer.* Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 *Drying tube.* To contain approximately 300 g of silica gel.

5.3.3 *Calibration gas.* Refer to paragraph 6.1.

5.3.4 *Filter.* As recommended by NDIR manufacturer.

5.3.5 CO_2 *removal tube.* To contain approximately 500 g of ascarite.

5.3.6 *Ice water bath.* For ascarite and silica gel tubes.

5.3.7 *Valve.* Needle valve, or equivalent, to adjust flow rate

5.3.8 *Rate meter.* Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min. (0.035 cfm) through NDIR.

5.3.9 *Recorder (optional).* To provide permanent record of NDIR readings.

6. *Reagents.*

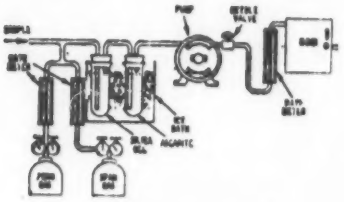

Figure 10-3. Analytical equipment.

6.1 *Calibration gases.* Known concentration of CO in nitrogen (N_2) for instrument span, prepurified grade of N_2 for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within ±2 percent of the specified concentration.

6.2 *Silica gel.* Indicating type, 6 to 16 mesh, dried at 175° C (347° F) for 2 hours.

6.3 *Ascarite.* Commercially available.

7. *Procedure.*

7.1 *Sampling.*

7.1.1 *Continuous sampling.* Set up the equipment as shown in Figure 10-1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See § 7.2 and 8). CO_2 content of the gas may be determined by using the Method 3 integrated sample procedure (36 FR 24886), or by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 *Integrated sampling.* Evacuate the flexible bag. Set up the equipment as shown in Figure 10-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak free. Sample at a rate proportional to the stack velocity. CO_2 content of the gas may be determined by using the Method 3 integrated sample procedures (36 FR 24886), or by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

7.2 *CO Analysis.* Assemble the apparatus as shown in Figure 10-3, calibrate the instrument, and perform other required operations as described in paragraph 8. Purge analyzer with N_2 prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10-1.

8. *Calibration.* Assemble the apparatus according to Figure 10-3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of one hour for warm-up. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, ar i CO_2 removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

TABLE 10-1.—*Field data*

Location ---------- Comments:
Test ----------
Date ----------
Operator ----------

Clock time	*Rotameter setting, liters per minute (cubic feet per minute)*

9. *Calculation—Concentration of carbon monoxide.* Calculate the concentration of carbon monoxide in the stack using equation 10-1.

$$C_{CO_{stack}} = C_{CO_{NDIR}}(1 - F_{CO_2}) \qquad \text{equation 10-1}$$

where:

$C_{CO_{stack}}$ = concentration of CO in stack, ppm by volume (dry basis).

$C_{CO_{NDIR}}$ = concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).

F_{CO_2} = volume fraction of CO_2 in sample, i.e., percent CO_2 from Orsat analysis divided by 100.

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APPENDIX

A. Performance Specifications for NDIR Carbon Monoxide Analyzers.

Range (minimum)	0–1000ppm.
Output (minimum)	0–10mV.
Minimum detectable sensitivity	20 ppm.
Rise time, 90 percent (maximum)	30 seconds.

ENDIX B

A flow chart of the operations involved in an auditing program, fro
it setting desired limits on the data quality to filing the results,
en in the following pages. Assumed numbers are used and a sample
culation of an audit is performed in the flow chart. Each operation
erences to the section in the text of the report where it is discusse

MANAGER

1. LIMITS FOR DATA QUALITY CAN BE SET BY WHAT IS DESIRED OR FROM THE NATURAL VARIABILITY OF THE METHOD WHEN USED BY TRAINED AND COMPETENT PERSONNEL. FOR THIS EXAMPLE, IT IS ASSUMED THAT $\sigma_R\{CO\}$ = 43.5 ppm (subsec.4.1), AND USING + 3 $\sigma_R\{CO\}$, THE LIMITS ARE L = -131 ppm AND U = 131 ppm.

2. FROM PRIOR KNOWLEDGE OF DATA QUALITY, ESTIMATE THE PERCENTAGE OF FIELD MEASUREMENTS FALLING OUTSIDE THE ABOVE LIMITS. IF NO INFORMATION IS AVAILABLE, MAKE AN EDUCATED GUESS. IT IS ASSUMED IN THIS EXAMPLE THAT 50 PERCENT OF THE FIELD DATA ARE OUTSIDE THE LIMITS L AND U (subsec. 4.4.4).

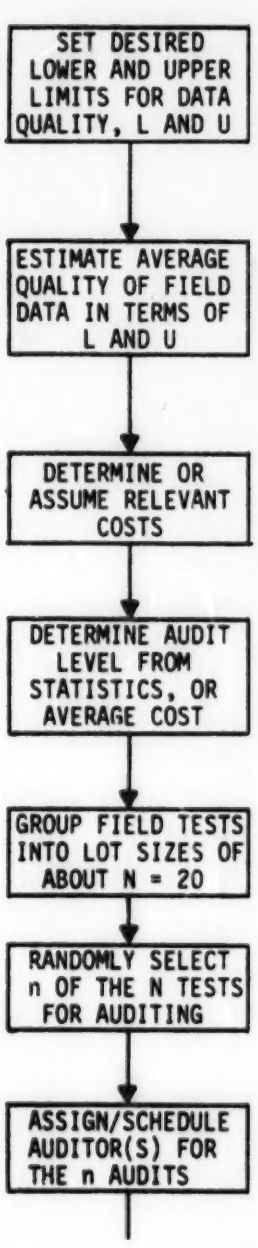
3. DETERMINE: (1) COST OF CONDUCTING AN AUDIT, (2) COST OF FALSELY INFERRING THAT GOOD DATA ARE BAD, (3) COST OF FALSELY INFERRING THAT BAD DATA ARE GOOD, AND (4) COST SAVINGS FOR CORRECTLY IDENTIFYING BAD DATA (subsec. 4.4.4).

4. DETERMINE THE AUDIT LEVEL EITHER BY (1) MINIMIZING AVERAGE COST USING EQUATION (22) OF SUBSECTION 4.4.4, OR (2) ASSURING A DESIRED LEVEL OF CONFIDENCE IN THE REPORTED DATA THROUGH STATISTICS. FOR THIS EXAMPLE, THE AUDIT LEVEL IS TAKEN AS n = 5 (fig. 15).

5. BY TEAMS, TYPES OF SOURCES, OR GEOGRAPHY, GROUP FIELD TESTS INTO LOTS (GROUPS) OF ABOUT 20, TO BE PERFORMED IN A PERIOD OF ONE CALENDAR QUARTER.

6. SELECT n OF THE N TESTS FOR AUDITING. COMPLETE RANDOMIZATION MAY NOT BE POSSIBLE DUE TO AUDITOR'S SCHEDULE. THE PRIMARY POINT IS THAT THE FIELD TEAM SHOULD NOT KNOW IN ADVANCE THAT THEIR TEST IS TO BE AUDITED.

7. ASSIGN OR SCHEDULE AN AUDITOR FOR EACH FIELD TEST.


1
SET DESIRED LOWER AND UPPER LIMITS FOR DATA QUALITY, L AND U
2
ESTIMATE AVERAGE QUALITY OF FIELD DATA IN TERMS OF L AND U
3
DETERMINE OR ASSUME RELEVANT COSTS
4
DETERMINE AUDIT LEVEL FROM STATISTICS, OR AVERAGE COST
5
GROUP FIELD TESTS INTO LOT SIZES OF ABOUT N = 20
6
RANDOMLY SELECT n OF THE N TESTS FOR AUDITING
7
ASSIGN/SCHEDULE AUDITOR(S) FOR THE n AUDITS

AUDITOR

8. THE AUDITOR OBTAINS APPROPRIATE CALIBRATED EQUIPMENT AND SUPPLIES FOR THE AUDIT (subsec. 4.3).

9. OBSERVE THE FIELD TEAM'S PERFORMANCE OF THE FIELD TEST (subsec. 4.3.2) AND NOTE ANY UNUSUAL CONDITIONS THAT OCCURRED DURING THE TEST.

10. THE AUDITOR'S REPORT SHOULD INCLUDE (1) DATA SHEET FILLED OUT BY THE FIELD TEAM , (2) AUDITOR'S COMMENTS, (3) AUDIT DATA SHEET WITH CALCULATIONS , AND (4) A SUMMARY OF THE TEAM'S PERFORMANCE WITH A NUMERICAL RATING (subsec. 4.3).

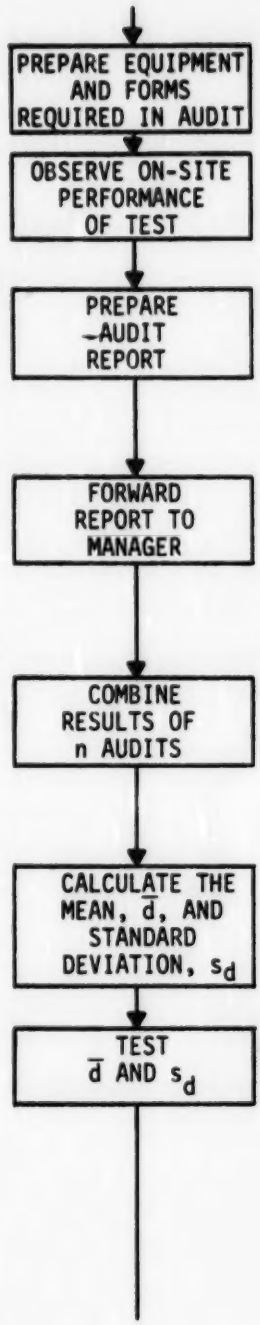
11. THE AUDITOR'S REPORT IS FORWARDED TO THE MANAGER.

MANAGER

12. COLLECT THE AUDITOR'S REPORTS FROM THE n AUDITS OF THE LOT OF N STACKS. IN THIS CASE n = 7 AND ASSUMED VALUES FOR THE AUDITS ARE d_1 = -40, d_2 = 20, d_3 = -10, d_4 = 80, d_5 = 60, d_6 = 30, and d_7 = 10 (table 4).

13. CALCULATE $\bar{d}$ AND s_d ACCORDING TO THE SAMPLE IN TABLE 4. RESULTS OF THIS SAMPLE CALCULATION SHOW $\bar{d}$ = 21.4 AND s_d = 40.6 (table 4, subsec. 4.4.3).

14. USE A t-TEST TO CHECK $\bar{d}$ FOR SIGNIFICANCE, FOR THIS EXAMPLE $t = (21.4 \times \sqrt{7})/43.5 = 1.30$. THE TABULATED t-VALUE FOR 6 DEGREES OF FREEDOM AT THE 0.05 LEVEL IS 1.943; HENCE, $\bar{d}$ IS NOT SIGNIFICANTLY DIFFERENT FROM 0 AT THIS LEVEL. ALSO, s_d IS CHECKED AGAINST THE ASSUMED VALUE OF 43.5 ppm BY A CHI-SQUARE TEST.
$\chi^2/f = s_d^2/\sigma^2\{\bar{d}\} = (40.6)^2/(43.5)^2 = 0.87$
THE TABULATED VALUE OF $\chi^2/6$ AT THE 95 PERCENT LEVEL IS 1.64; HENCE, s_d IS NOT SIGNIFICANTLY DIFFERENT FROM 43.5 ppm.



15. OBTAIN THE VALUE OF k FROM TABLE 5, FOR n = 7 AND p = 0.1. THIS VALUE IS 2.334, THEN $\bar{d} + k\, s_d$ = 116 ppm AND $\bar{d} - k\, s_d$ = -71.4ppm (subsec. 4.4.3).

16. COMPARE THE ABOVE CALCULATIONS WITH LIMITS L AND U (subsec. 4.4.3). FOR THIS EXAMPLE

$$\bar{d} + k\, s_d = 116 < U = 131 \text{ ppm}$$

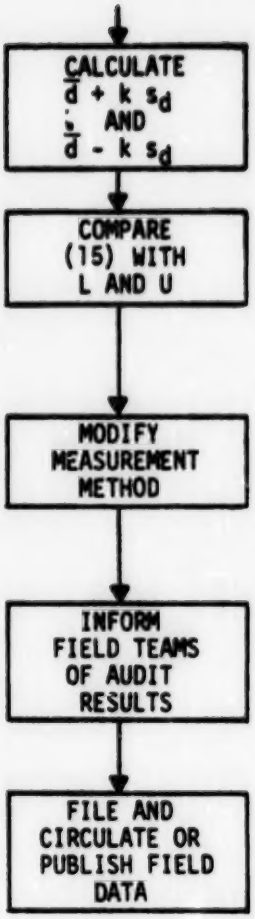
$$\bar{d} - k\, s_d = -71.4 > L = -131 \text{ ppm}$$

BOTH CONDITIONS ARE SATISFIED GO TO 18*.

17. STUDY THE AUDIT AND FIELD DATA FOR SPECIFIC AREAS OF VARIABILITY, SELECT THE MOST COST-EFFECTIVE ACTION OPTION(S) THAT WILL RESULT IN GOOD QUALITY DATA (subsec. 4.2). NOTIFY THE FIELD TEAMS TO IMPLEMENT THE SELECTED ACTION OPTION(S).

18. A COPY OF THE AUDITOR'S REPORT SHOULD BE SENT TO THE RESPECTIVE FIELD TEAM. ALSO, THE DATA ASSESSMENT RESULTS, i.e., CALCULATED VALUES OF $\bar{d}$, s_d, AND COMPARISON WITH THE LIMITS L AND U SHOULD BE FORWARDED TO EACH TEAM INVOLVED IN THE N FIELD TESTS.

19. THE FIELD DATA WITH AUDIT RESULTS ATTACHED ARE FILED. THE AUDIT DATA SHOULD REMAIN WITH THE FIELD DATA FOR ANY FUTURE USES.



*if either one or both limits had been exceeded one would proceed to step 17.

APPENDIX C GLOSSARY OF SYMBOLS

This is glossary of symbols as used in this document. Symbols used and defined in the reference method (appendix A) are not repeated here.

SYMBOL	DEFINITION
N	Lot size, i.e., the number of field tests to be treated as a group.
n	Sample size for the quality audit (section IV).
CV{X}	Assumed or known coefficient of variation (100 σ_X/μ_X).
$\hat{CV}\{X\}$	Computed coefficient of variation (100 $s_X/\bar{X}$) from a finite sample of measurements.
$\sigma\{X\}$	Assumed standard deviation of the parameter X (population standard deviation).
$\hat{\tau}\{X\}$	Computed bias of the parameter X for a finite sample (sample bias).
R	Range, i.e., the difference in the largest and smallest values in r replicate analyses.
d_j	The difference in the audit value and the value of CO arrived at by the field crew for the j^{th} audit.
$\bar{d}$	Mean difference between CO_j and CO_{aj} for n audits.
s_d	Computed standard deviation of differences between CO_j and CO_{aj}.
p	Percent of measurements outside specified limits L and U.
k	Constant used in sampling by variables (section IV).
P{Y}	Probability of event Y occurring.
$t_{(n-1)}$	Statistic used to determine if the sample bias, $\bar{d}$, is significantly different from zero (t-test).
$\chi^2/(n-1)$	Statistic used to determine if the sample variance, s^2, is significantly different from the assumed variance, σ^2, of the parent distribution (chi-square test).

APPENDIX C GLOSSARY OF SYMBOLS (CONTINUED)

SYMBOL	DEFINITION
L	Lower quality limit used in sampling by variables.
U	Upper quality limit used in sampling by variables.
CL	Center line of a quality control chart.
LCL	Lower control limit of a quality control chart.
UCL	Upper control limit of a quality control chart.
CO	Carbon monoxide reported by the field team for field test.
CO_a	Carbon monoxide concentration used in an audit check.
CO_m	Measured value of a calibration gas.
CO_t	Assayed or known value of a calibration gas.
$C_{CO_{NDIR}}$	Concentration of CO measured by the NDIR analyzer on a dry basis and uncorrected for CO_2 removal.

APPENDIX D GLOSSARY OF TERMS

The following glossary lists and defines the statistical terms as used in this document.

Accuracy	A measure of the error of a process expressed as a comparison between the average of the measured values and the true or accepted value. It is a function of precision and bias.
Bias	The systematic or nonrandom component of measurement error.
Lot	A specified number of objects to be treated as a group, e.g., the number of field tests to be conducted by an organization during a specified period of time (usually a calendar quarter).
Measurement method	A set of procedures for making a measurement.
Measurement process	The process of making a measurement, including method, personnel, equipment, and environmental conditions.
Population	A large number of like objects (i.e., measurements, checks, etc.) from which the true mean and standard deviation can be deduced with a high degree of accuracy.
Precision	The degree of variation among successive, independent measurements (e.g., on a homogeneous material) under controlled conditions, and usually expressed as a standard deviation or as a coefficient of variation.
Quality audit	A management tool for independently assessing data quality.
Quality control check	Checks made by the field crew on certain items of equipment and procedures to assure data of good quality.
Sample	Objects drawn, usually at random, from the lot for checking or auditing purposes.

APPENDIX E CONVERSION FACTORS

Conversion factors for converting the U.S. customary units to the International System of Units (SI)* are given below.

To Convert from	To	Multiply by
	Length	
foot	meter (m)	0.3048
inch	meter (m)	0.0254
	Pressure	
inch of mercury (in. of Hg) (32°F)	Newton/meter^2 (N/m^2)	3386.389
inch of mercury (in. of Hg) (60°F)	Newton/meter^2 (N/m^2)	3376.85
millimeter mercury (mmHg) (32°F)	Newton/meter^2 (N/m^2)	133.3224
inch of water (in. of H_2O) (29.2°F)	Newton/meter^2 (N/m^2)	249.082
inch of water (in. of H_2O) (60°F)	Newton/meter^2 (N/m^2)	248.84
	Force	
pound-force (lbf avoirdupois)	Newton (n)	4.448222
	Mass	
pound-mass (lbm avoirdupois)	kilogram (kg)	0.4535924
	Temperature	
degree Celsius	kelvin (K)	$t_K = t_C + 273.15$
degree fahrenheit	kelvin (K)	$t_K = (t_F+459.67)/1.8$
degree rankine	kelvin (K)	$t_K = t_R/1.8$
degree fahrenheit	degree Celsius	$t_C =)t_F - 32)/1.8$
kelvin	degree Celsius	$t_C = t_K - 273.15$
	Velocity	
foot/second	meter/second (m/s)	0.3048
foot/minute	meter/second (m/s)	0.00508
	Volume	
cubic foot (ft^3)	meter^3 (m^3)	0.02832
	Volume/Time	
foot^3/minute	meter^3/second (m^3/s)	0.0004719
foot^3/second	meter^3/second (m^3/s)	0.02832

*Metric Practice Guide (A Guide to the Use of SI, the International Systems of Units), American National Standard Z210.1-1971, American Society for Testing and Materials, ASTM Designation:E380-70, Philadelphia, Pa., 1971.